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DEVELOPMENT OF A LABORATORY TEST FOR MULTIPOINT INJECTOR
DEPOSITS: APPROACHES 1 AND 2(U) SOUTHWEST RESEARCH INST
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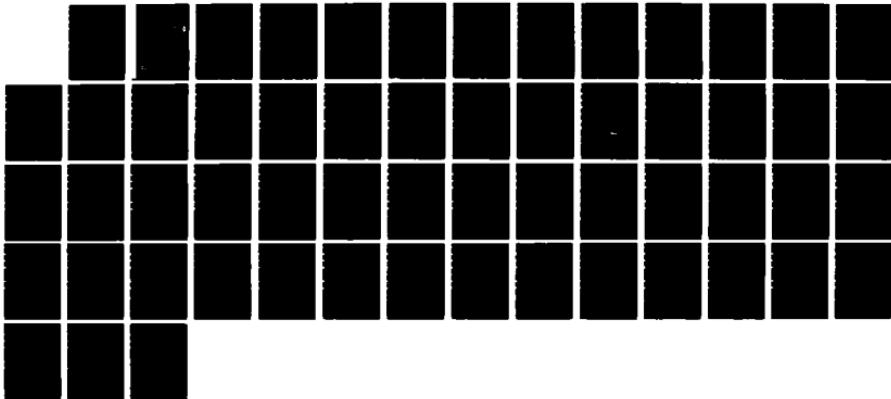
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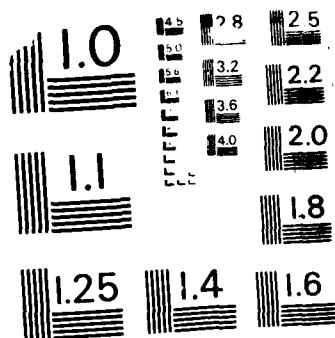
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MICROCOPY RESOLUTION TEST CHART
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DEVELOPMENT OF A LABORATORY TEST FOR MULTIPORT INJECTOR DEPOSITS: APPROACHES I AND II

AD-A190687

FINAL REPORT

No. SwRI-4765/1

Prepared by

**L.L. Stavinoha and R.M. Estefan
H.W. Marbach, Jr.
Southwest Research Institute
6220 Culebra Road
San Antonio, Texas 78284**

A large, bold, black stamp. The word "DTIC" is at the top in a large, sans-serif font. Below it, the word "ELECTED" is written in a slightly smaller font. Underneath that, the date "JAN 28 1988" is stamped. On the far left is a large, stylized letter "S", and on the far right is a large, stylized letter "D". A small pair of scissors icon is positioned between the "S" and the "D".

Prepared for

**The Coordinating Research Council, Inc.
219 Perimeter Parkway
Atlanta, Georgia 30346**

Contract No. CM-128-85 (1-86)

September 1987



SOUTHWEST RESEARCH INSTITUTE
SAN ANTONIO HOUSTON

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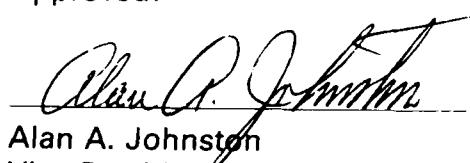
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Alan A. Johnston
Vice-President
Fuels and Lubricants Research Division

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Technical discussions and recommendations provided by John Bowden and Edward Dimitroff during the progress of this program are hereby gratefully acknowledged.



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I. GENERAL BACKGROUND AND APPROACH TO METHOD DEVELOPMENT

A. Introduction and Background

A number of current production automobiles equipped with Multiport Fuel Injector (MPFI) delivery systems are encountering injector restriction or "plugging," caused by a buildup of fuel-related deposits. The buildup of these deposits takes place predominately on the pintle tip protruding from the injector.

Injector plugging can occur with as little as 1,000 miles of short-trip urban driving, resulting in poor vehicle drivability, hard starting, reduced fuel economy, and loss of power. In severe cases, the vehicle is rendered inoperative. The problem is geographically widespread. Thus far, the principal contributing factors (other than fuel and heat during engine shutdown) have not been definitely identified. Industry experience and preliminary testing have shown that engine design, location and size of injectors, fuel/air distribution, various organic species in fuel, the chemical and physical properties of gasolines, and numerous other parameters may contribute to the rate and severity of deposit buildup.

Past studies^{(1-6)*} have shown that the following conditions exist and may either hinder or accelerate deposition:

- The most successful driving cycle to optimize injector plugging appears to be a 15-minute operating period followed by a 45-minute "soak" period. Heat soak-back during shutdown appears to be critical.
- Temperatures in the injectors average about 100° to 120°F during operation and reach 210° to 290°F during the 45-minute soak period.
- Injectors are determined to be plugged if the flow rate is restricted by 10 percent or more.

*Underscored numbers in parentheses refer to the List of References in Section IV of this report.

- Injector fouling results from a deposit buildup that occurs primarily on the pintle protruding from the nozzle, especially in the critical area of the metering orifice, resulting in reduced fuel flow and an erratic spray pattern. The pintle is made of 440C stainless steel.
- Injectors with low weep rates foul more readily than tight closures. Those injectors with high weep rates tend not to foul.
- It is generally agreed that certain fuel detergents in sufficient concentration can alleviate or prevent injector plugging.

These deposits can cause significant performance problems. Thus, there is an immediate need for a laboratory bench test that can screen possible problem fuels and also permit future deposit-inhibiting additives to be evaluated prior to full-scale fleet studies. The results of this bench test must be reproducible, relatively rapid, and correlate with in-service MPFI performance in vehicle tests.

At least one bench test currently exists for evaluation of deposit-forming tendencies of fuels in spark-ignition engines. This procedure, the Induction System Deposit (or ISD) test, was developed approximately 20 years ago by the U. S. Army at Southwest Research Institute (SwRI) for screening fuels with a tendency to form intake valve deposits.⁽⁷⁾

A second-generation apparatus, referred to as gasoline dispersant/detergent additive evaluation (GD/DAE) bench rig, was also developed by the U. S. Army at SwRI to evaluate both intake valve and carburetor deposits simultaneously as well as additive effectiveness in controlling these deposit formations.⁽⁸⁾

These tests, along with others, have been used to study deposition or additive effectiveness and have been modified in numerous ways by individuals interested in a specific condition, additive, fuel, or application. However, published literature has not identified any specific modifications or new procedures that can accurately predict

gasoline multiport fuel injector deposit formation tendencies or relate bench scale-produced deposits with real vehicle operational results.

SwRI proposed two different approaches, to be performed in parallel, that would permit the contacting of fuel to a heated metal surface under varying conditions. Either approach could result in the desired goal of CRC Project No. CM-128 to develop a bench test to determine fuel/fuel-additive tendencies to produce injector deposits.

Approach I would evaluate a number of modifications to the GD/DTE rig described in SAE Paper No. 790204, February 26, 1979.⁽⁸⁾ The device would provide a means of fuel, air, blowby, and deposit surface variations, all of which may play a major role in the deposition process.

Approach II would evaluate a closed system in which heated fuel materials in a vapor state can interact with each other and with fuel on a hot metal surface, a process that may contribute significantly to the deposition process. This report covers both Approaches I and II and are discussed in separate sections.

B. Program Objective

The objective of this program was to develop a laboratory bench test for screening gasolines to determine their potential for forming deposits in automotive multiport fuel injectors (MPFI). The test to be developed must be repeatable, reproducible, and correlatable with fuel injector performance in vehicle tests.

It was anticipated that both proposed laboratory approaches would simulate the conditions leading to MPFI deposit formation in vehicles. These conditions would include atmospheric pressure, repetitious cycling, including fuel exposure followed by a hot-soak up to 210° to 290°F, and test specimen material of 440C stainless steel. These test conditions did not exclude other possibilities, since the ultimate requirement was a laboratory test that correlates with vehicle test results.

Additional requirements were for the bench test to be completed in an 8-hour period or less, a reasonably small test sample, allowance for the measurement of deposit

formation, and finally, discrimination between fuels with and without detergent additives. Again, the proposed systems would be designed to meet these requirements.

C. Base Test Fuels

Three test fuels (coded A, B, and C in Table 1) were employed in this program.

TABLE 1. TEST FUELS FOR MPFI GASOLINE
DETERGENCY TEST DEVELOPMENT

<u>Fuel Code</u>	<u>Anticipated Deposit Level</u>	<u>Description</u>
A	High	Ashland MPI Batch No. 4
B	Intermediate	Phillips J
C	Low	Indolene Clear

Test fuels A and B in Table 1 were used initially to develop laboratory test conditions. After the conditions were optimized, fuels A and C were to be tested a minimum of three times to provide a measure of laboratory test repeatability.

Tables 2 to 4 summarize characterization data developed for test fuels A, B, and C, respectively. Note low induction period (ASTM D 525) of fuel A and high (4 hr) oxidation deposit levels (ASTM D 873), indicating potential poor storage stability. Different code numbers were assigned to each drum of test fuel A. While fuel A drums 15707 and 15732 were opened in January 1987, drum 15733 was not opened until September 1987. Each fuel was blanketed with argon and maintained at 38°F.

D. Additive-Treated Test Fuels

A CRC letter (dated 27 February 1987) stated:

"The following approach to choosing additives for the fuels in the fuel injector deposit program was agreed to at the February 26 group meeting:

- Each company interested in submitting an additive will send one candidate additive to SwRI. The supplier is to submit one gallon of the additive.

TABLE 2. RESULTS OF CHARACTERIZATION OF TEST FUEL A

<u>Code No.</u>	15707-G A <u>1-13-87</u>	15707-G A <u>3-24-87</u>	15732-G A <u>1-16-87</u>	15733-G A <u>9-10-87</u>	15977-G A* <u>4-7-87</u>
<u>Test Fuel</u>					
<u>Date of Analyses</u>					
Induction Period, D 525, minutes	240	195	225	--	225
Unwashed Gum, D 381, mg/100 mL	7.3	7.2	5.8	--	11.0
Existen Gum, D 381, mg/100 mL	4.3	6.8	4.0	--	9.7
Proposed Method for Oxidation					
Stability, D 873,					
4-hour Total Unwashed Gum, mg/100 mL**	173.7	--	157.8	--	157.8
4-Hour Total Washed Gum, mg/100 mL**	169.3	--	157.5	--	130.9
Induction System Deposit, Fed.					
Test Method 500.1,					
300 mL, 300°F, mg/300 mL	3.4	5.7	--	1.3	--
300 mL, 375°F, mg/300 mL	2.9	4.5	3.7	--	3.4
300 mL, 400°F, mg/300 mL	2.9	--	--	--	--
300 mL, 275°F, mg/300 mL	--	--	--	2.3	6.3
Induction System Deposit, Fed.					
Test Method 500.1,					
100 mL, 300°F, mg/100 mL	0.8	--	--	--	--
100 mL, 375°F, mg/100 mL	0.9	--	1.2	--	--
100 mL, 400°F, mg/100 mL	0.9	--	--	--	--
FIA					
● Aromatics, D 1319, vol%	30.7	--	--	--	--
● Olefins, D 1319, vol%	15.0	--	--	--	--
● Saturates, D 1319, vol%	54.3	--	--	--	--

* Drum stored at ambient under cover since October 1986.

** Average of duplicate runs.

- Accompanying each submission of a candidate additive will be sufficient documentation to demonstrate that vehicle test results can discriminate between fuels with and without the additive. There must be similar documentation to show that a concentration less than the optimum level recommended by the supplier will result in an increased deposit-forming tendency.
- Each company submitting an additive must indicate whether the additive is in category A, an amine-type detergent; or category B, a polymeric-type dispersant.
- SwRI is to assign a number to each additive submitted and, after a two-week period starting from February 26th, choose two additives from category A and two from category B. Selection is to be made randomly by generation of random numbers that relate to the numbers assigned. SwRI will not reveal which additives have been selected to any of the participants and will not disclose which companies sent in additives.

TABLE 3. RESULTS OF CHARACTERIZATION
OF TEST FUEL B

<u>Code No.</u>	15734-G
<u>Test Fuel</u>	B
Induction Period, D 525, minutes	>1440
Unwashed Gum, D 381, mg/100 mL	2.2
Existent Gum, D 381, mg/100 mL	1.1
Proposed Method for Oxidation Stability, D 873:	
4-hour Total Unwashed Gum, mg/100 mL*	3.1
4-hour Total Washed Gum, mg/100 mL*	1.5
Induction System Deposit, Federal Test Method 500.1, 375°F	
300 mL, mg/300 mL	1.6
100 mL, mg/100 mL	0.1
Aromatics, D 1319, vol%	38.4
Olefins, D 1319, vol%	15.2
Saturates, D 1319, vol%	46.4

*Average of duplicate runs

TABLE 4. RESULTS OF CHARACTERIZATION
OF TEST FUEL C

<u>Code No.</u>	15929-G
<u>Test Fuel</u>	C
Induction Period, D 525, minutes	>960
20 psi drop point, minutes	555
Unwashed Gum, D 381, mg/100 mL	2.0
Existent Gum, D 381, mg/100 mL	1.2
Induction System Deposit, Federal Test Method 500.1	
300 mL, 300°F, mg/300 mL	1.0
300 mL, 375°F, mg/300 mL	1.4
FIA	
Aromatics, D 1319, vol%	26.9
Olefins, D 1319, vol%	3.9
Saturates, D 1319, vol%	69.2

- SwRI will blend the four selected additives into the Ashland fuel (the greater deposit-forming fuel of the two current fuels), at the concentration recommended by the supplier, and proceed with the small-scale device tests. In addition, a suitable quantity of each of the four additive-containing fuels will be sent to Pittsburgh Applied Research Center for testing in the JFTOT. PARC's address is:

Pittsburgh Applied Research Corporation
 c/o Dr. Harold O. Strange
 University of Pittsburgh Applied Research Center
 100 William Pitt Way
 Pittsburgh, Pennsylvania 15238

CRC will solicit bids to perform vehicle tests in the fuels with the selected additives. Consequently, it may be desirable to blend sufficient test fuel for a vehicle test up to 6000 miles on each additive-containing fuel."

Table 5 summarizes the additive-treated test fuel codes, laboratory-assigned code number, date prepared, and description for four additives (W through Z) at two concentrations (H=high, L=low).

In May 1987, 4.5 gallons of each additive test fuel was prepared (using fuel A from drum 15732-G) in 5-gallon metal containers. Then, two gallon sample cans were filled from the stock containers for laboratory testing and one set for shipment to Pittsburgh Applied Research Corporation.

TABLE 5. ADDITIVE-TREATED TEST FUEL

Fuel Code	Laboratory Code Number	Date Prepared	Description		
			Base Fuel	Additive Code	Additive Concentration
XWH	16037-G	May 28, 1987	Ashland	W	Clean up; H=High
XWL	16038-G	May 28, 1987	Ashland	W	Keep clean; L=Low
XEH	16039-G	May 12, 1987	Ashland	X	Clean up; H=High
XEL	16040-G	May 12, 1987	Ashland	X	Keep clean; L=Low
XYH	16041-G	May 12, 1987	Ashland	Y	Clean up; H=High
XYL	16042-G	May 12, 1987	Ashland	Y	Keep clean; L=Low
ZEH	16043-G	May 12, 1987	Ashland	Z	Clean up; H=High
ZEL	16044-G	May 12, 1987	Ashland	Z	Keep clean; L=Low

II. APPROACH I: GASOLINE DETERGENCY TEST (GDT)

A. General

The apparatus described in SAE Paper No. 790204 was developed specifically to examine fuel deposition tendencies in carburetor throat areas and intake valves. Therefore, the conditions established for use in the test procedure were tailored to two different environments, neither of which may be directly related to fuel injection tip deposits. However, the apparatus does lend itself to multiple variations in fuel, air, reaction gas species, and changes in deposit surface metallurgy and temperature conditions. This built-in flexibility and the considerable experience gained in its use provided a good starting apparatus in the development of a usable MPFI deposit evaluation procedure.

SwRI proposed to operate the apparatus shown in Figure 1 under a number of varying conditions to seek an optimum procedure. These variations included:

1. Changing the deposit tube from aluminum to 440C material.
2. Varying the deposit tube temperature from a steady-state heated condition to a cold tube upon which fuel is sprayed, followed by a heating period that simulates "hot soak" without fuel. This is accomplished by spraying fuel on a hot deposit tube, followed by cooling to room temperature, and then washing with n-heptane (or test fuel), followed by oven-heating prior to weighing.
3. Determining by the presence and absence of different blowby contaminants if these materials alter the deposition process. The original procedure used only the aqueous part of blowby; however, the gas and/or organic portion could be evaluated if necessary to determine hot temperature deposit effects.
4. Delivering small amounts of fuel (weeping) onto the tube surface during the hot soak period simulation, which is accomplished by varying fuel spray rate onto the heated deposit tube.
5. Varying the fuel wetting and hot soak periods to enhance final deposit values. This procedure is accomplished by varying the time duration during which fuel is sprayed onto the hot deposit surface.

6. Varying air flow rate during the hot soak period to determine any effects due to rapid vapor removal from around the deposit surface.
7. Varying fuel washing action over the deposit surface.
8. Though the present procedure has a cold carburetor sleeve area, this part of the apparatus will not be used in these studies.
9. The highest deposit fuel (to be provided by CRC and identified as fuel A) would be used for all preliminary evaluations. When conditions are found to be conducive to reasonable deposit buildup with reasonable repeatability, the low deposit fuel C (as well as fuel B) would be evaluated to determine the degree of discrimination. As discrimination is established, the remaining fuels in the CRC schedule would be run to establish fuel-ranking capabilities.
10. Since the rig is simple, and it is anticipated that tests can be shorter than 8 hours in duration, the many variations described above, plus others that may evolve, can be rapidly accomplished early in the program. This would permit the researchers an opportunity to optimize the procedure within the project time period, in the hope that this apparatus or some variation thereof could produce the desired results.

B. Discussion

The gasoline detergent/dispersant additive evaluation (GD/DAE) apparatus in Figure 1 and on the left in Figure 2 was developed specifically to evaluate the fuel-depositing tendencies in both cold carburetor throat and hot intake valve areas. Experience gained in-house at SwRI suggested removal of the carburetor sleeve section to run the tests in the Gasoline Detergency Test (GDT) configuration at the right in Figure 2 for this program. For these preliminary tests, the "cone" sprayer was maintained in the test apparatus, recognizing that at a later date it may be advantageous to replace it with a "flat" sprayer.

The starting parameters were:

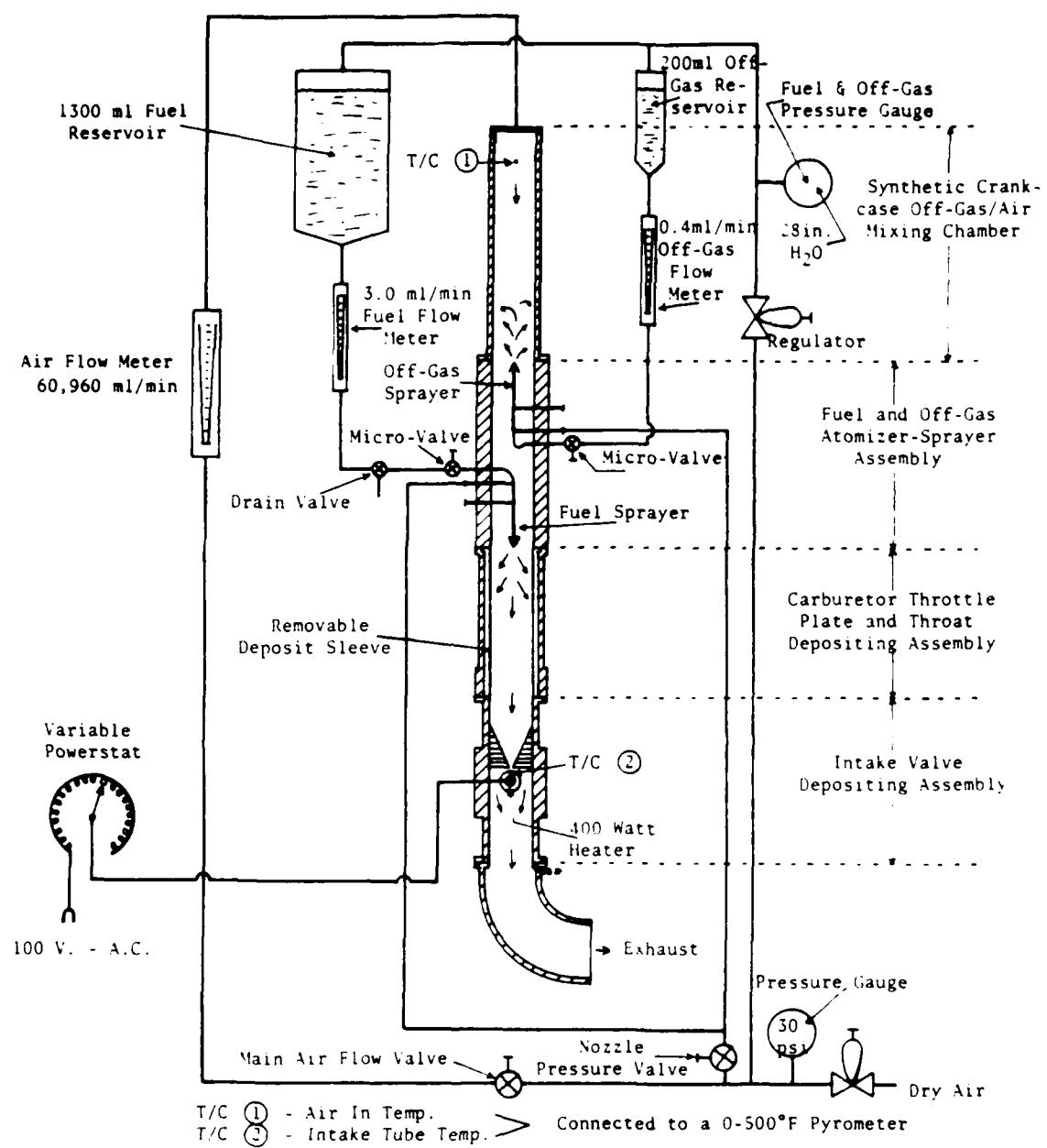


FIGURE 1. CROSS-SECTION AND FLOW DIAGRAM OF THE
DISPERSANT/DETERGENT ADDITIVE EVALUATION BENCH RIG

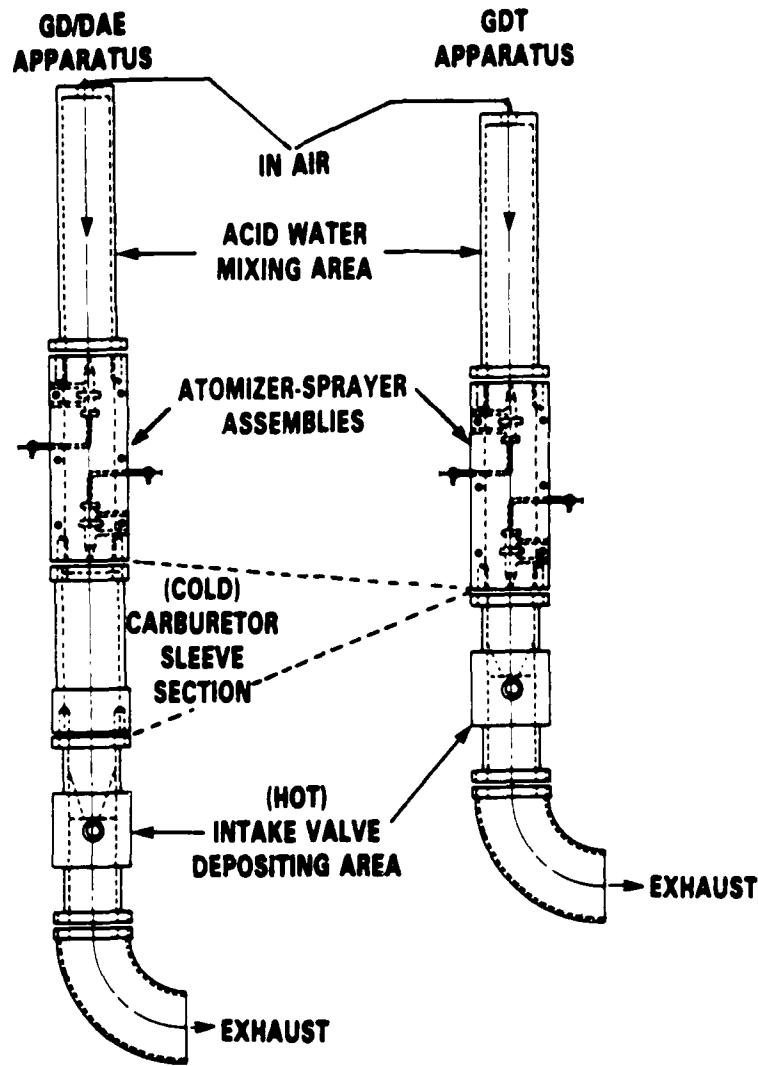
1. The dry main air flow enters the top of the apparatus at a flow rate of 61.0 L/min.
2. The acid water flow at a rate of 0.4 mL/min was sprayed (7.4 L/min air atomizer flow) against the air flow through an atomizer-sprayer and was regulated by a microvalve.
3. The test fuel flow at a rate of 3 mL/min was sprayed (through an atomizer-sprayer) with air flow (7.4 L/min) and was also regulated by a microvalve.
4. The metal deposit collection tube was heated to selected temperatures by a 0.5-inch diameter bar heater inserted into the deposit tube. The temperature was measured by a thermocouple in the upper part of the deposit tube and controlled by a manual voltage regulator. Details of apparatus preparation operation, deposit tube preparation, handling and weighing, etc., can be provided.

At the outset of this optimization phase, two basic criteria were established for the GDT apparatus test tube deposits. These criteria were that,

1. Fuel A, a high-depositing fuel (Ashland Batch No. 4), have tube deposits greater than 10 mg, and that
2. Fuel B, a intermediate-depositing fuel (Phillips J), have tube deposits approximately 1/4 to 1/3 of those of fuel A.

It is noted that fuel C (added later in the program) was to be considered a "cleaner" fuel.

The two fuels were evaluated at deposit tube temperatures of 250°, 300°, and 350°F. In addition, aluminum and stainless steel were evaluated with both the high- and intermediate-depositing fuels. The acid water was used only at (1) the standard dilute mixture of hydrochloric, nitric, and sulfuric acid at the flow rate of 0.4 mL/min.



- FIGURE 2. GD/DAE AND GDT CONFIGURATIONS

and (2) without acid water for both high- and intermediate-depositing fuels. Finally, the main air flow was varied from the standard 61.0 L/min to 30.5, 15.2 L/min, and eventually 0 L/min.

Data for this phase of the program are summarized as Test Nos. 22-1 through 22-25 in Table 6. Test Nos. 22-26 through 22-36 represent a second phase of optimization in which both acid water and air to the acid nozzle (7.5 L/min) were shut off to seek optimum conditions for test time, fuel flow rate, and main air flow. Air flow from the fuel nozzle was maintained (in all tests) at 7.5 L/min.

TABLE 6. GDT EVALUATIONS

Test No.	Test Fuel	Test Date	Tube Temp., °F	Test Time, hr	Fuel Volume, L	Fuel Flow, mL/min	Acid Flow, mL/min	Main Air Flow, L/min	Tube Metal*	Tube Deposit, mg		Normalized mg/L Unwashed	Normalized mg/L Washed
										Unwashed	Washed		
22-01	A	1-02-87	300	6	1.072	3.0	0.4	61.0	AL	7.4	5.3	6.9	4.9
22-02	A	1-05-87	350	6	1.069	3.0	0.4	61.0	AL	9.2	8.4	8.6	7.9
22-03	A	1-06-87	250	6	1.088	3.0	0.4	61.0	AL	10.0	4.0	9.2	3.7
22-04	A	1-07-87	300	6	1.079	3.0	0.4	61.0	SS**	12.8	11.7	11.9	10.8
22-05	A	1-08-87	350	6	1.063	3.0	0.4	61.0	SS**	9.4	8.8	8.8	8.3
22-06	A	1-12-87	250	6	1.090	3.0	0.4	61.0	SS**	25.1	4.5	23.0	4.1
22-07	B	1-15-87	300	6	1.084	3.0	0.4	61.0	AL	4.0	3.0	3.7	2.8
22-08	B	1-16-87	300	6	1.119	3.0	0.4	61.0	SS	5.1	3.5	4.6	3.1
22-09	A	1-21-87	300	6	1.080	3.0	0.4	61.0	SS	(0)	0	0	0
22-10	A	1-23-87	300	6	1.068	3.0	0.4	61.0	AL	14.4	11.6	13.5	10.9
22-11	A	1-26-87	300	6	1.093	3.0	0.4	61.0	SS	12.6	10.9	11.5	10.0
22-12	B	1-27-87	350	6	1.108	3.0	0.4	61.0	AL	6.9	4.7	6.2	4.2
22-13	A	1-28-87	300	6	1.120	3.0	0.0	61.0	AL	15.9	13.2	14.2	11.8
22-14	A	1-29-87	300	6	1.106	3.0	0.0	30.5	AL	21.9	15.7	19.8	14.2
22-15	B	1-30-87	250	6	1.130	3.0	0.4	61.0	AL	5.2	2.4	4.6	2.1
22-16	A	2-02-87	300	6	1.120	3.0	0.4	30.5	AL	13.0	8.3	11.6	7.4
22-17	A	2-03-87	300	6	0.769	2.2	0.0	30.5	AL	5.9	4.8	7.7	6.2
22-18	A	2-04-87	300	6	1.081	3.0	0.0	15.2	AL	25.9	18.2	24.0	16.8
22-19	A	2-05-87	300	6	1.126	3.0	0.4	61.0	AL	9.3	8.3	8.3	7.4
22-20	B	2-06-87	300	6	1.077	3.0	0.0	15.2	AL	13.9	5.8	12.9	5.4
22-21	A	2-12-87	300	6	1.103	3.0	0.4	61.0	AL	9.1	7.9	8.3	7.2
22-22	A	2-16-87	300	6	1.152	3.0	0.4	30.5	AL	7.1	5.6	6.2	4.9
22-23	A	2-17-87	280	6	1.136	3.0	0.4	61.0	AL	11.3	9.5	9.9	8.4
22-24	A	2-20-87	300	6	1.083	3.0	0.4***	61.0	AL	11.7	10.5	10.8	9.7
22-25	A	2-23-87	300	3	0.719	4.0	0.0	15.2	AL	17.1	13.2	23.8	18.4
22-26	A	3-22-87	300	3	0.725	4.0	0.0****	0.0	AL	47.5	--	65.5	--
22-27	A	3-23-87	300	3	0.600	3.0	0.4****	0.0	AL	30.3	20.2	33.7	30.0
22-28	A	3-23-87	300	2*	0.243	2.0	0.0****	0.0	AL	10.2	7.3	42.0	30.0
22-29	A	3-24-87	300	2	0.378	3.0	0.0****	15.2	AL	9.3	6.3	24.6	16.7
22-30	A	3-24-87	300	2	0.247	2.0	0.0****	15.2	AL	2.8	2.7	11.3	10.9
22-31	A	3-26-87	300	2	0.371	3.0	0.0****	30.5	AL	5.1	4.4	13.7	11.9
22-32	B	3-26-87	300	2	0.345	3.0	0.0****	30.5	AL	1.2	0.5	3.5	1.5
22-33	A	3-27-87	300	3	0.722	4.0	0.0****	30.5	AL	13.0	11.2	18.0	15.5
22-34	A	3-27-87	300	2	0.470	4.0	0.0****	30.5	AL	8.2	6.8	17.4	14.5
22-35	B	3-31-87	300	3	0.710	4.0	0.0****	30.5	AL	5.2	3.2	7.3	4.5
22-36	C	3-31-87	300	3	0.730	4.0	0.0****	30.5	AL	5.3	2.3	3.2	3.2

* AL = aluminum, SS = stainless steel 400C.

** 25 microinch surface finish, remainder with 15 microinch surface finish.

*** Deionized water only, no acid.

**** No air flow from acid water nozzle.

***** 6.8 L/min fuel nozzle air flow. (7.5 L/min is normal air flow at fuel nozzle.)

Figure 3 shows the tube deposition of the high-depositing fuel A, using both stainless steel and aluminum deposit tubes, at three different deposit tube temperatures of 250°, 300°, and 350°F and a fuel flow of 3.0 mL/min with acid water flow of 0.4 mL/min. The highest optimum deposit was produced at 300°F with no apparent catalytic effect between the aluminum or stainless steel deposit tubes. In addition, good reproducibility was accomplished for two tests at 300°F.

The intermediate-depositing test fuel B was also evaluated at 250°, 300°, and 350°F (Figure 4) with a fuel flow of 3.0 mL/min and acid water flow of 0.4 mL/min. The stainless steel deposit tube was only used at the 300°F temperature because of the initial results for fuel A, which showed no metallurgical effects. The results from these data appear to be linear with temperature with the highest deposit at 350°F.

The difference between fuel A and fuel B deposit levels at 300°F met the initial criteria set for this phase. Additionally, the difference between stainless steel and aluminum deposit tubes appeared to be insignificant. Therefore, because of the economics involved in their manufacture and the deposit-to-tube weight ratio, the aluminum deposit tube was selected for the majority of the remaining work.

The original purpose of the acid water was to simulate the engine off-gases that contributed to formation of carburetor throat deposits. Because the carburetor sleeve section has been removed for MPFI testing, the acid water flow was also stopped to evaluate its effect. Data from Table 6 were selected to demonstrate the effect of main air flow on fuel A depositing tendencies at 300°F with and without acid water in Figure 5, which suggests:

1. The lower the main air flow, the higher the deposit level, and
2. Acid water flow at 0.4 mL/min at a nozzle air flow of 7.5 L/min tends toward lower deposit levels at a main air flow of 30.5 L/min compared to 61 L/min.

NOTE: In this and subsequent figures, deposit values used are mg/L calculated from the test fuel volume and deposit weight.

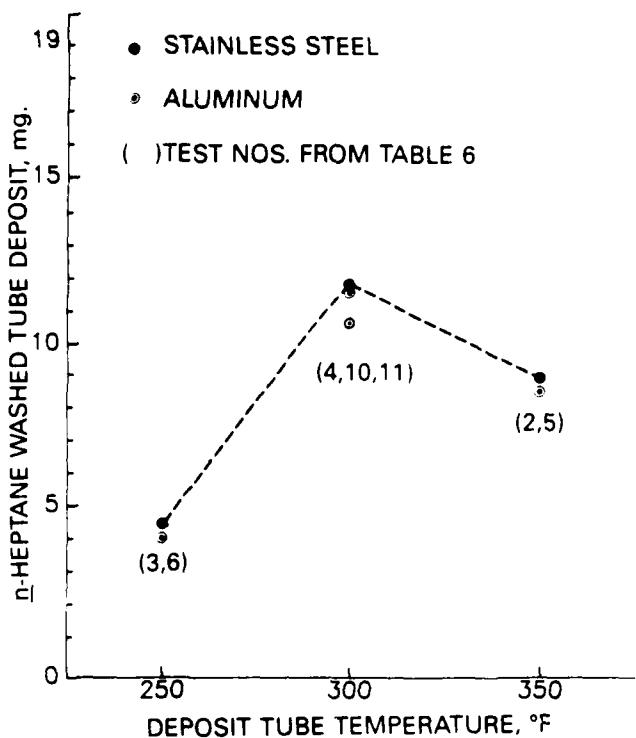


FIGURE 3. HIGH-DEPOSITING FUEL A AT THREE TEMPERATURES USING STAINLESS STEEL AND ALUMINUM DEPOSIT TUBES WITH ACID

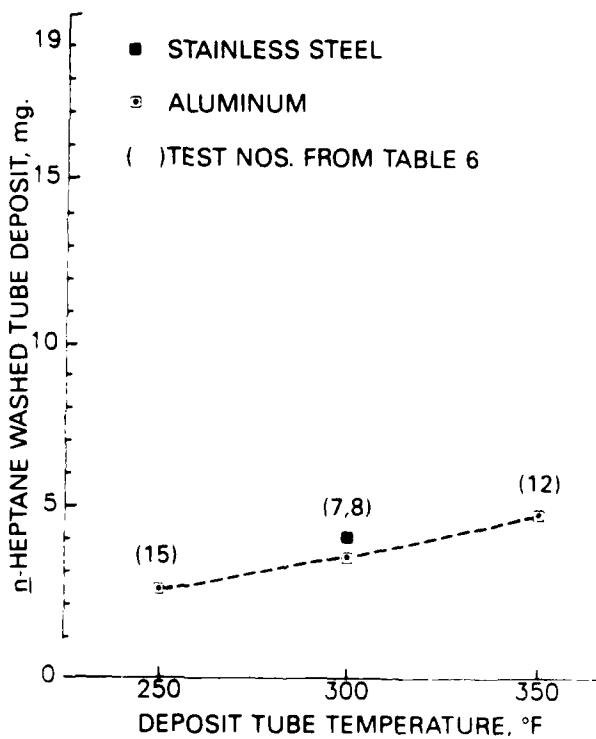


FIGURE 4. LOW-DEPOSITING FUEL B AT VARIOUS TEMPERATURES WITH ACID

3. Based on limited data, variation of fuel flow rate from 3 to 4 mL/min seemed to have a minor effect on deposit level, while the 2 mL/min rate seemed less repeatable and gave somewhat lower values.

Based on the favorable absolute deposit values of 11.2 mg (14.5 mg/L) and 3.2 mg (4.5 mg/L) (test Nos. 22-33 and 22-35) for test fuels A and B, respectively, which used 30.5 L/min main air flow and no acid water, the initial absolute target values were met, and the following conditions were selected (as optimized with respect to deposit formation) for repeatability testing:

- Test time set at 3 hours (fuel A gave > 10 mg for ease of weighing)
- Fuel flow at 4.0 mL/min (tube did not appear flooded; deposit greater for low time with high fuel flow, i.e., greater than 3 mL/min)

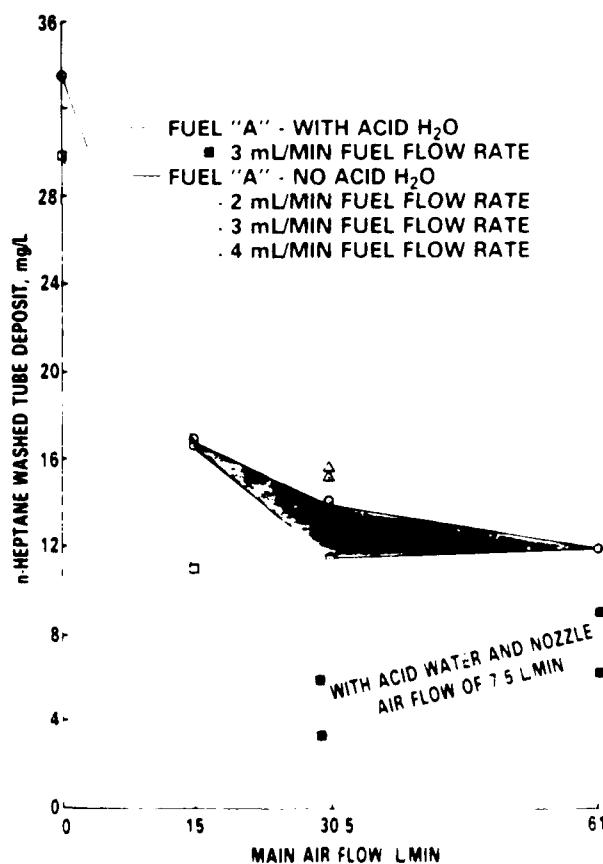


FIGURE 5. EFFECT OF MAIN AIR FLOW ON FUEL A AT 300°F
WITH AND WITHOUT ACID WATER FLOW

- Test tube temperature at 300°F (lower temperature required lower flow rate for good deposit behavior)
- Main air flow at 30.5 L/min (lower air flow rates gave deposits all over tube with "run-out" of sleeves)
- No acid water or nozzle air flow.

Under these conditions, fuel C (test No. 22-36) gave an absolute value of 2.3 mg (or 3.2 mg/L).

Table 7 is a listing of data for test runs beginning with test 22-37. Table 8 (based on data from runs 22-37 to 22-68) provides a summary of average tube deposit values for test fuels A, B, and C and four additives (W, X, Y, and Z) at two concentrations (H and L abbreviated for "clean up" and "keep clean," respectively). These data suggest that additive X is very effective while W is also very effective at the H concentration; while X is better than W at the L (keep clean) concentration. Both additives Y and Z produced (n-heptane insoluble) deposits, which were higher in magnitude than that obtained for the base fuel A. This is more vividly demonstrated in Figure 6, which also contains data for test fuels B, BYH, BZH, C, and CYH. Additive fuel BYH gave very high deposits when compared to both base fuel B and additive fuel BZH, which were essentially equivalent. Also included in Table 8 are calculated standard deviation values that indicate the repeatability of the deposit values.

Test Nos. 22-81 through 22-84 using "clean" test fuel C and additive Y at the H (clean up) concentration suggested that the Y additive was not forming deposits in and of itself at either 250° or 300°F.

Since all four additives evaluated in this program were stated to be effective in engine tests at optimum concentrations (H and L), conditions were sought under which additives Y and Z would appear effective deposit modifiers.

TABLE 7. GDT EVALUATIONS

Test No.	Fuel No.	Fuel Code	Tube Metal*	Tube Temp., °C	Main Air Flow, L/min	Fuel Flow, ml/min	Fuel Volume, ml		Tube Deposit, mg/L		Normalized, mg/L Unwashed	Normalized, mg/L Washed
							Unwashed	Washed	Unwashed	Washed		
22-37	15732-C	A	AL	300	30.5	4.0	735	15.5	12.6	21.0	17.1	13.6
22-38	15732-C	A	AL	300	30.5	4.0	705	13.1	9.6	18.9	13.6	13.5
22-39	15732-C	A	AL	300	30.5	4.0	705	13.4	9.5	19.0	13.6	13.5
22-40	15929-C	C	AL	300	30.5	4.0	660	2.8	1.3	4.2	2.0	1.4
22-41	15929-C	C	AL	300	30.5	4.0	720	3.0	1.0	4.2	1.4	1.4
22-42	15929-C	C	AL	300	30.5	4.0	705	3.5	1.2	5.0	1.7	1.7
22-44	15734-C	B	AL	300	30.5	4.0	710	6.9	2.6	9.7	3.7	3.7
22-45	15734-C	B	AL	300	30.5	4.0	710	6.2	2.5	8.7	3.5	3.5
16039-C	AYH	AL	300	30.5	4.0	760	13.5	0.4	44.1	0.5	0.5	
16039-C	AXH	AL	300	30.5	4.0	710	35.0	0.8	49.3	1.1	1.1	
16039-C	AXH	AL	300	30.5	4.0	760	31.3	0.6	41.2	0.8	0.8	
16040-C	AXL	AL	300	30.5	4.0	710	18.7	1.3	26.3	1.8	1.8	
16040-C	AXL	AL	300	30.5	4.0	750	18.4	1.3	24.5	2.0	2.0	
16040-C	AXL	AL	300	30.5	4.0	720	21.2	2.5	29.4	3.2	3.2	
16040-C	AXL	AL	300	30.5	4.0	750	15.7	2.5	35.5	20.9	20.9	
16041-C	AYH	AL	300	30.5	4.0	740	29.6	26.0	40.0	32.4	32.4	
16041-C	AYH	AL	300	30.5	4.0	750	25.1	19.3	33.3	26.3	26.3	
16042-C	AYL	AL	300	30.5	4.0	735	18.9	1.3	25.7	20.8	20.8	
16042-C	AYL	AL	300	30.5	4.0	730	14.7	12.4	20.1	17.0	17.0	
16042-C	AYL	AL	300	30.5	4.0	715	18.2	14.7	25.5	20.6	20.6	
16043-C	AZH	AL	300	30.5	4.0	720	21.2	13.3	29.4	19.2	19.2	
16043-C	AZH	AL	300	30.5	4.0	740	19.7	16.4	26.6	19.5	19.5	
16043-C	AZH	AL	300	30.5	4.0	700	19.6	13.4	28.0	19.1	19.1	
16044-C	AYH	AL	250	30.5	4.0	710	33.0	2.3	46.5	3.2	3.2	
16044-C	AYH	AL	250	30.5	4.0	720	45.5	3.1	63.2	4.3	4.3	
16044-C	AYH	AL	300	30.5	4.0	720	32.9	1.2	45.7	1.7	1.7	
16037-C	AWH	AL	300	30.5	4.0	720	34.5	1.4	47.9	1.9	1.9	
16037-C	AWH	AL	300	30.5	4.0	720	34.7	1.3	48.2	1.8	1.8	
16038-C	AWL	AL	300	30.5	4.0	720	23.1	5.3	32.1	7.4	7.4	
16038-C	AWL	AL	300	30.5	4.0	710	23.9	5.2	33.7	7.3	7.3	
16038-C	AWL	AL	300	30.5	4.0	728	24.7	2.4	33.9	3.2	3.2	
16038-C	AWL	AL	300	30.5	4.0	715	30.0	5.0	42.0	7.0	7.0	
16044-C	AZL	AL	250	30.5	4.0	715	30.5	4.9	42.7	6.9	6.9	
16044-C	AZL	AL	250	30.5	4.0	720	20.0	0.9	27.8	1.3	1.3	
15732-C	A	AL	250	30.5	4.0	720	12.7	8.2	17.6	11.5	11.5	
15732-C	A	AL	250	30.5	4.0	720	42.4	1.6	58.9	2.2	2.2	
16039-C	AYH	AL	250	30.5	4.0	723	24.7	4.3	36.2	5.9	5.9	
16039-C	AYH	AL	350	30.5	4.0	708	40.3	0.9	56.9	1.3	1.3	
16037-C	AWH	AL	250	30.5	4.0	715	30.5	4.9	42.7	6.9	6.9	
16037-C	AWH	AL	350	30.5	4.0	710	18.9	8.7	26.6	12.3	12.3	
15732-C	A	AL	250	30.5	4.0	460	14.8	3.7	32.2	8.0	8.0	
15732-C	A	AL	250	30.5	4.0	525	5.2	1.8	9.9	3.4	3.4	
16041-C	AYH	AL	250	30.5	4.0	540	10.2	6.3	18.9	11.7	11.7	
16041-C	AYH	AL	350	30.5	4.0	540	7.8	3.4	14.4	6.3	6.3	
16037-C	AWH	AL	250	30.5	4.0	592	5.7	0.7	9.6	1.2	1.2	
16037-C	AWH	AL	350	30.5	4.0	705	9.8	1.4	13.9	2.0	2.0	
15732-C	A	AL	250	30.5	4.0	560	5.5	0.9	9.5	ND	ND	
15732-C	A	AL	250	30.5	4.0	720	15.3	1.3	21.3	1.8	1.8	
16041-C	BYH	AL	300	30.5	4.0	715	13.6	10.7	19.0	15.0	15.0	
16041-C	BYH	AL	250	30.5	4.0	540	4.2	0.6	7.8	1.1	1.1	
16235-C	CYH	AL	300	30.5	4.0	690	11.2	2.1	16.2	3.0	3.0	
16235-C	CYH	AL	300	30.5	4.0	540	3.6	8.7	6.7	0.4	0.4	
16235-C	CYH	SS	61.0	3.0	0.0	546	ND*	ND	ND	26.7	26.7	
16235-C	CYH	SS	30.0	0.0	0.0	530	ND	ND	ND	7.5	7.5	
16231-C	BYH	AL	250	30.5	4.0	730	46.0	5.7	63.0	7.8	7.8	
16231-C	BYH	AL	250	30.5	4.0	540	ND	1.3	ND	2.4	2.4	
16231-C	BYH	AL	300	30.5	4.0	525	ND	1.0	ND	1.9	1.9	
16232-C	B2H	AL	250	30.5	4.0	720	11.7	10.2	16.3	14.1	14.1	
16232-C	B2H	AL	300	30.5	4.0	725	9.6	9.3	13.2	12.8	12.8	
22-87	16041-C	AYH	AL	250	30.5	4.0	710	18.9	13.4	27.8	18.0	18.0
22-88	16041-C	AYH	AL	300	30.5	4.0	525	5.2	1.8	9.9	3.4	3.4
22-89	16041-C	AYH	AL	300	30.5	4.0	730	10.2	6.3	18.9	11.7	11.7
22-90	16041-C	AYH	AL	300	30.5	4.0	540	4.2	0.6	7.8	1.1	1.1
22-91	15732-C	A	AL	250	30.5	4.0	730	46.0	5.7	63.0	7.8	7.8
22-92	16041-C	AYH	AL	250	30.5	4.0	525	ND	1.3	ND	2.4	2.4
22-93	16041-C	AYH	AL	250	30.5	4.0	720	11.7	10.2	16.3	14.1	14.1
22-94	16379-C	(SWRI)	AL	300	30.5	4.0	725	9.6	9.3	13.2	12.8	12.8
22-95	16661-G	(EG&G)	AL	300	30.5	4.0	725	9.6	9.3	13.2	12.8	12.8

* AL : aluminum, SS : stainless steel 400C.

† ND : Not Determined; tube washed with test fuel followed by n-heptane and oven drying for 1 hour at 230°C.

TABLE 8. SUMMARY OF AVERAGE TUBE DEPOSITS FOR TEST FUEL
CODES A, B, C AND FOUR ADDITIVES IN TEST FUEL A
AT TWO CONCENTRATIONS

Fuel Code	Tube Deposit, mg/L		
	Unwashed	Washed	Standard Deviation
	Average*	Average*	
A	19.6	14.9	1.7
B	9.2	3.6**	0.1
C	4.5	1.7	0.2
AWH	47.3	1.8	0.1
AWL	33.2	6.0	1.9
AXH	44.9	0.8	0.2
AXL	26.7	2.4	0.8
AYH	36.3	26.6	4.7
AYL	23.8	19.5	1.8
AZH	28.0	19.3	0.2
AZL	NR	NR	NR

NR - Not Run

* - Average of Three Tests

** - Average of Two Tests

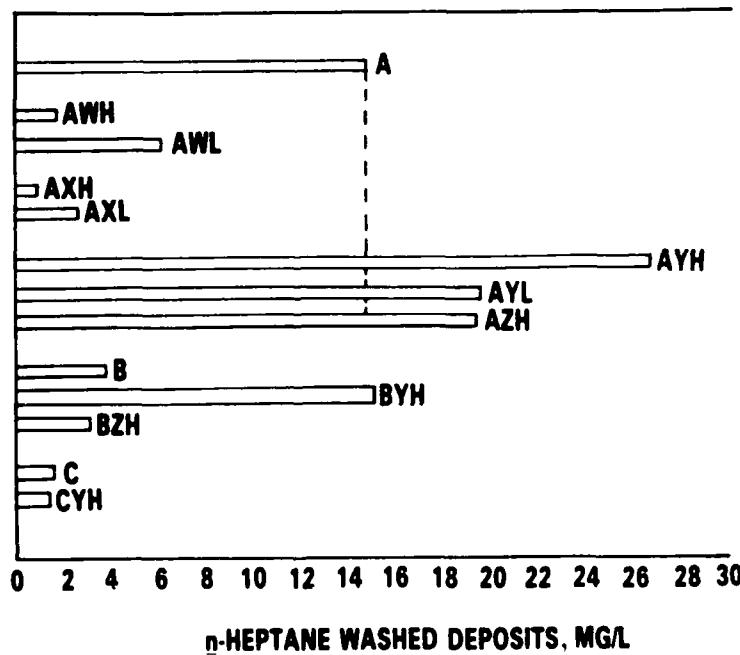


FIGURE 6. 300°F OPTIMIZED CONDITIONS

Table 9 summarizes test Nos. 22-61, 22-62, 22-69, 22-70, 22-71, and 22-91, which were run to see if additives Y and Z were effective at 250°F (with all other parameters the same as at 300°F). While the A deposit was very low (7.8 mg/L compared to 14.7 mg/L at 300°F), both AYH and AZH additives gave lower deposits (3.2 and 4.3 mg/L, respectively), but AZL had little or no effect (6.9 and 7.0 mg/L).

Data for reduced fuel flow rate (3 mL/min) at high main air flow rate (61 L/min) for base fuel A and additive-treated fuels AYH and AZH showed an increase in deposits over fuel A (test Nos. 22-78, 22-79, and 22-80).

Figure 7 summarizes data for test fuel A and additive fuels AWH, AXH, AYH, and AZH at various temperatures using the "optimized" test conditions of 30.5 L/min main air, zero acid water/zero acid water nozzle air flow, and a fuel flow rate of 4 mL/min for 3-hour test periods. Note the effectiveness of additives W and X at both 250° and 300°F compared to fuel A deposits. At 350°F, the efficacy of additive W is equivalent to the base fuel A while X is 50 percent efficient in deposit reduction; additives Y and Z show approximately 50 percent efficiency in deposit reduction at 250°F, but result in higher deposit levels at 300°F compared to the base test fuel A.

TABLE 9. OPTIMIZATION AT 250°F FOR FUEL A

Fuel Code	Test No.	Fuel Flow Rate	Airflow Rate	Tube Deposit, mg/L	
				Unwashed	Washed
A	22-91	4	30.5	63.0	7.8
A	22-71	4	30.5	27.8	1.3?*
AZH	22-62	4	30.5	63.2	4.3
AZL	22-69	4	30.5	42.0	7.0
AZL	22-70	4	30.5	42.7	6.9
AYH	22-61	4	30.5	46.5	3.2
A	22-77	3	30.5	32.2	8.0
A	22-78	3	61.0	9.9	3.4
AYH	22-79	3	61.0	18.9	11.7
AZH	22-80	3	61.0	14.4	6.3

*? considered low outlier based on deposit appearance that looked heavier.

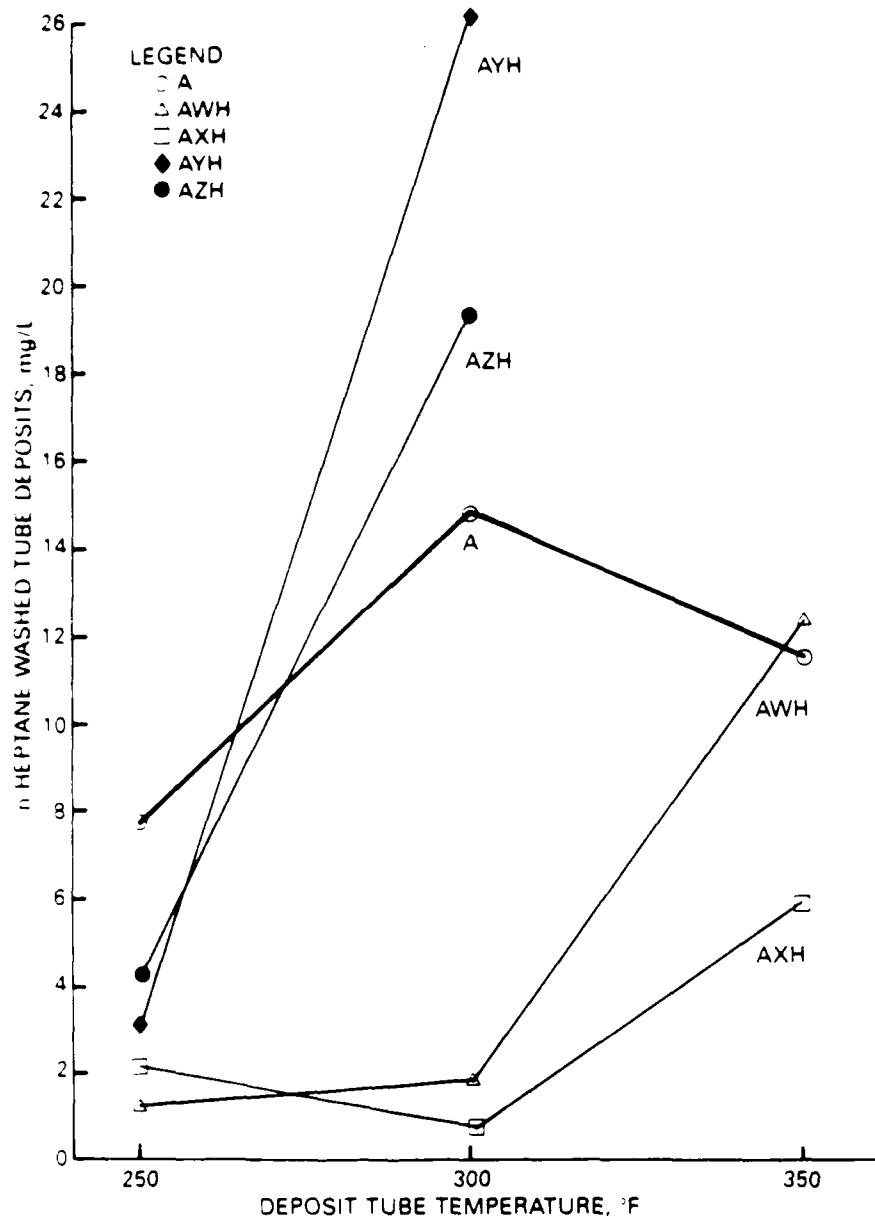


FIGURE 7. EFFECT OF TEMPERATURE ON TUBE DEPOSITS
(OPTIMIZED CONDITIONS)

Data for test Nos. 22-89 and 22-90 were superimposed on Figure 5 to provide the comparison in Figure 8, which implies that reduced air flow results in the additive Y becoming effective in giving reduced deposits (7.8 mg/L for AYH versus approximately 30 mg/L for fuel A at 0 main air flow rate). This observation is reinforced in Figure 8, which also shows a value of 500 mg/L for fuel A based on data generated in Approach II. Approach II uses the reactor with hot soak airflow rates of 2 L/min, which is much lower than the total air flow rate achievable with the GDT apparatus.

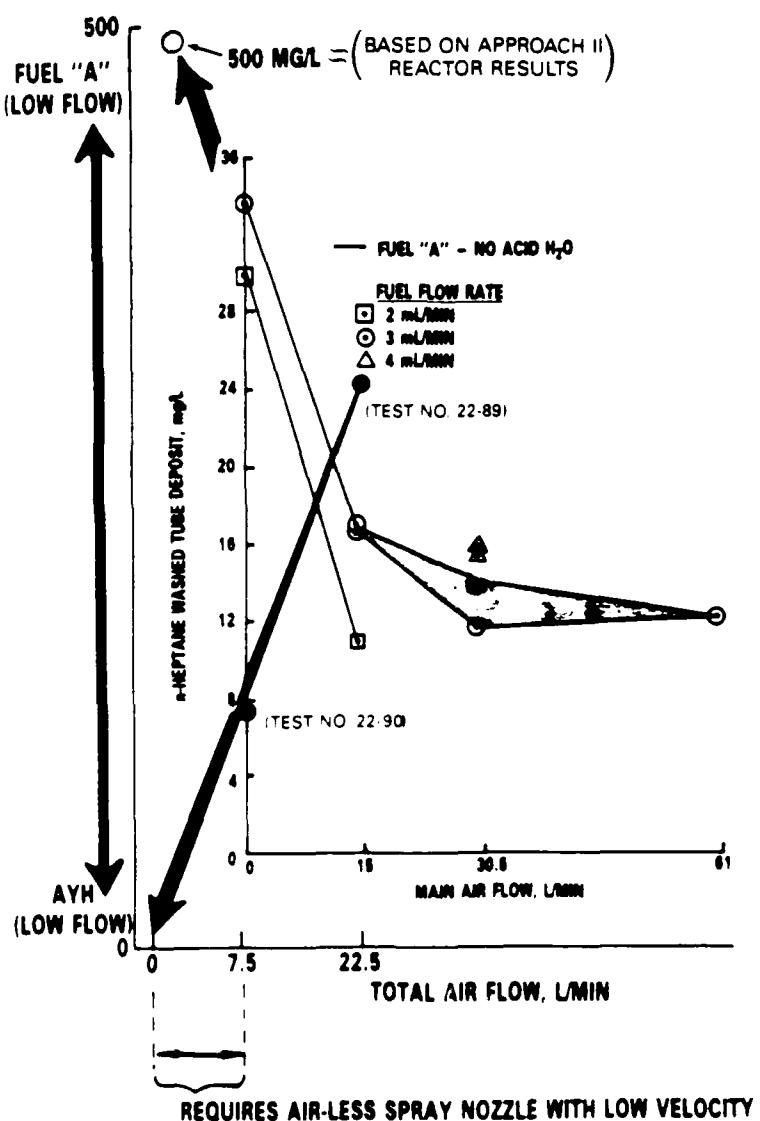


FIGURE 8. EFFECT OF TOTAL AIR FLOW ON FUEL A AND AYH DEPOSIT LEVELS

The GDT apparatus design, i.e., cone spray with 7.5 L/min air nebulizer nozzle and venturi baffles ahead of deposit tube, does not lend itself to "good" operation at 0 main airflow rates nor to reduced nebulizer spray nozzle airflow rates below 7.5 L/min. Additionally, good spray development and fuel flow control seem to be best attained at 3 to 4 mL/min, but not much below 3 mL/min.

Since the effectiveness of additives Y and Z appear to be linked to their inherent volatility, which would then be affected by tube test temperature, air transfer (flow rate), deposit exposure time, deposit tube-handling conditions (e.g., oven drying before washing), etc., it is implied in Figure 8 that an "airless" spray nozzle apparatus may ultimately be employed with very low main-air flow to properly rate the effectiveness (of this type of additive's ability) to modify MPI-type deposits. Such an apparatus does not now exist and would require development. Thus, it was decided to explore the "additive volatility theme" by performing a limited matrix of tests using ISD apparatus. It should be noted that additive thermal or oxidative stability may be the dominating mechanism, rather than (or in conjunction with) volatility. This mechanism would effect bench test deposit levels for these additives; hence the choice of the term "Additive Volatility Theme."

The Induction System Deposit (ISD) Method,⁽⁷⁾ Federal Test Method 500.1 of Federal Standard 791C,⁽⁹⁾ utilizes a flat spray (air-nebulizer) nozzle and is operated at a fuel flow rate of 2.0 mL/min with a nozzle air flow rate of 11.8 L/min. Figure 9 is a schematic representation of the ISD apparatus. Better sensitivity and repeatability can be

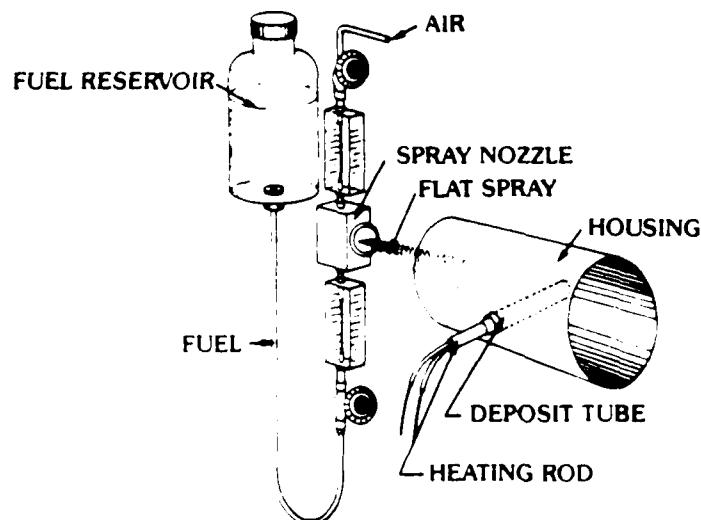


FIGURE 9. SCHEMATIC REPRESENTATION OF INDUCTION SYSTEM DEPOSIT (ISD) APPARATUS (FED. TEST METHOD 500.1)

achieved with this apparatus when larger fuel volumes are used as opposed to the 100 mL of fuel called for in Method 500.1. Using 300 mL of fuel sprayed at 2 mL/min onto a tube at 300°F, followed by 30 minutes of 230°F oven-drying of the deposit tube prior to fuel and n-heptane washing, the data in Table 10 were generated. In Table 10, it is noted that additives Y and Z are much less effective than W and X, and both AYL and AZL gave higher deposit levels than fuel A. Additives W and X produced very low values (0.7 and 0.3 mg/L, respectively) at the H concentration. AWL and AXL were not run, but would be expected to show a degree of effectiveness. Note the directionally lower value obtained for fuel B (1.0 mg/L) compared to fuel A (8.0 mg/L).

To further explore the volatility theme, modified ISD tests (performed without drying the deposit tube after the 150-minute fuel spray period) have qualitatively and quantitatively provided visual and gravimetric data that show additives Y and Z to be effective in test fuel A at the H concentration (see Table 11). For data in Table 11,

TABLE 10. RESULTS OF INDUCTION SYSTEM DEPOSIT TEST
(with Post Test Oven Heating)
July 20-28, 1987

Test Fuel	Tube Temp, °F	Fuel Volume, mL	Tube Deposit Washed,* mg/300 mL	Tube Deposit Normalized, mg/L
A	300	300	2.4**	8.0**
(A)	(300)	(300)	(2.9)	(9.7)***
AWH	300	300	0.2	0.7
AYH	300	300	1.9	6.3
AXH	300	300	0.1	0.3
AZH	300	300	2.2	7.3
B	300	300	0.3	1.0
AYL	300	300	6.3	21.0
50% AYL + 50% A	300	300	4.8	16.0
AZL	300	300	3.2	10.7
50% AZL + 50% A	300	300	3.2	10.7

* Tube oven-dried for 30 minutes at 230°F, cooled for 1 hour, then washed with test fuel, followed by n-heptane rinse and oven-drying for 30 minutes at 230°F.

** Value is lower than an expected value of approximately 5 mg/300 mL or 17 mg/L based on data in Table 2.

*** Parentheses () same as *, but only n-heptane washed. July 28, 1987

300 mL of fuel were used (at 2 mL/min on a 300°F tube), followed by a 3-minute cool down period in the ISD test rig and 5 to 10 minutes at room temperature. The deposit tube was then immersed in stirred fuel for 4 minutes and sequentially rinsed in two beakers of n-heptane to remove fuel. The tube was then oven dried for 30 minutes at 230°F, cooled for 30 minutes at room temperature, placed in a desiccator for 2 hours minimum and then weighed. In addition to quantitative data in Table 11, the behaviors of the deposit in the fuel and n-heptane wash were observed. Fuel A deposits appeared to be insoluble, but flaking from the tube surface was observed. Deposits from fuels AYH and AZH simply dissolved from the tube surface with no observable flaking. Deposits from AYL neither flaked nor dissolved. These results had been observed qualitatively previously, but they are in Table 11 supported by quantitative results. It has been speculated that the high value for fuel A (50.0 mg/L as opposed to an expected value of 15 to 20 mg/L) may have been enhanced by washing in fuel as opposed to just n-heptane, wherein absorption of fuel A into its deposit occurs and is not rinsed out by the n-heptane rinsing. It is more likely that the laboratory sample had deteriorated.

On September 10, 1987, it was learned that the CRC car test on test fuel A at EG&G had resulted in severe MPI fouling. Tests of bulk fuel samples from SwRI and EG&G (test run codes CRC-5 and CRC-13) showed low ISD values of 1.6 and 1.0 mg/300 mL, respectively. However, these values compared favorably with the 1.3 mg/300 mL value for fuel A from a freshly opened drum coded 15733 (Table 2). Evaluation of additive Y in fuel A (from drum 15733) at 275°F qualitatively modified the deposit, making it fuel-washable, but gave quantitative deposit weights essentially equal to the base fuel (test runs CRC-11 and CRC-12) when washed in test fuel.

The test deposit levels data (in Table 11) at 275° were higher than at 300°F (for drum 15733 and the EG&G fuel). However, the data were still much lower than the 13-15 mg/L range in the GDT runs 22-37 through 22-39, 22-94, and 22-95 (Table 7). To increase the deposit level, the ISD tube holder was placed in a vertical position for downward spraying onto the tube, as is the case with the GDT. After running several tests (run codes CRC-15 through CRC-18), the conditions of 275°F tube temperature with a spray nozzle air flow of 6 L/min was chosen for limited additive testing. Fuel flow rate in all runs was maintained at 2 mL/min. These data (CRC-19 through CRC-22)

TABLE II. RESULTS OF INDUCTION SYSTEM DEPOSIT TEST (WITHOUT POST-TEST OVEN HEATING)
(August 17 through September 19)

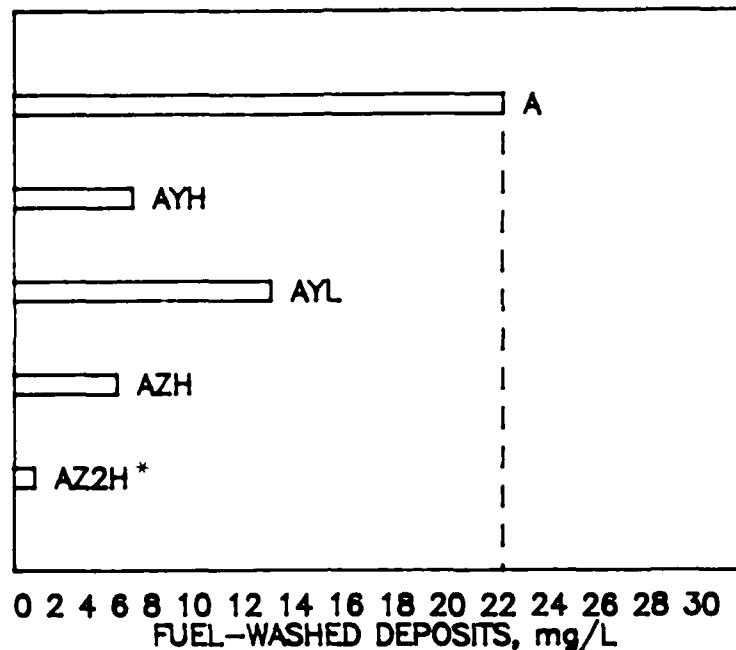
Date	Run Code	Fuel Code	Fuel Description	Tube Temp., °F		Air Flow, L/min	Fuel Vol., mL	Tube Deposit, mg/300 mL		Tube Deposit, mg/L	
				Unwashed	Washed			Unwashed	Washed	Normalized	Washed
08-17-87	CRC-1	16041	A YH	300	12	300	ND*	1.3 (F) ***	ND	4.3	ND
08-17-87	CRC-2	16042	A YL	300	12	300	ND	13.6 (F)	ND	45.3	ND
08-17-87	CRC-3	16043	A ZH	300	12	300	ND	1.5 (F)	ND	5.0	ND
08-17-87	CRC-4	15732	A	300	12	300	ND	15.0 (F) ***	ND	50.0	ND
09-10-87	CRC-5	16579	A (6000 gal)	300	12	300	ND	1.6 (C7) ***	ND	5.3	ND
09-10-87	CRC-6	16579	A (6000 gal)	290	12	300	ND	1.8 (C7)	ND	6.0	ND
09-10-87	CRC-7	16579	A (6000 gal)	290	9	300	ND	1.4 (C7)	ND	4.7	ND
09-11-87	CRC-8	15733	A (New Drum)	300	12	300	ND	1.3 (C7)	ND	4.3	ND
09-14-87	CRC-9	15732	A	300	12	300	ND	3.4 (C7)	ND	11.3	ND
09-14-87	CRC-10	15733 +	A YH (Fresh)	300	12	300	ND	6.4 (C7)	ND	21.3	ND
09-14-87	CRC-11	15733 +	A YH (Fresh)	275	12	300	ND	2.9 (F)	ND	9.7	ND
09-14-87	CRC-12	15733	A (New Drum)	275	12	300	ND	2.3 (F)	ND	7.7	ND
09-18-87	CRC-13	16661	A (EG&G)	300	12	300	ND	1.0 (C7)	ND	3.3	ND
09-18-87	CRC-14	16661	A (EG&G)	275	12	300	ND	2.2 (C7)	ND	7.3	ND
ISD: Vertical Configuration											
09-26-87	CRC-15	16661	A (EG&G)	275	12	300	ND	6.9 (F)	ND	23.0	ND
09-27-87	CRC-16	16661	A (EG&G)	275	4.5	300	ND	8.3 (F)	ND	27.7	ND
09-27-87	CRC-17	16661	A (EG&G)	250	6	200	ND	1.5 (F)	ND	7.5	ND
09-27-87	CRC-18	16661	A (EG&G)	275	6	300	ND	6.7 (F)	ND	22.3	ND
09-28-87	CRC-19	15733 +	A YH	275	6	300	ND	2.0 (F)	ND	6.7	ND
09-28-87	CRC-20	15733 +	A YL	275	6	300	ND	3.6 (F)	ND	12.0	ND
09-28-87	CRC-21	16661 +	A ZH	275	6	300	ND	1.7 (F)	ND	5.7	ND
09-29-87	CRC-22	16661 +	A + (2)ZH	275	6	300	ND	0.2 (F:AZH)	ND	0.7	ND

* ND = Not Determined.

** Fuel Rinse.

*** Value considered higher than 5 mg/300 mL expected value.

**** n-Heptane Rinse.



*AZ2H is "Z" at two times "H" concentration in fuel "A".

FIGURE 10. 275°F ISD CONDITIONS
(Data from Table 11)

are graphically summarized in Figure 10. The figure vividly demonstrates the ability of this modified ISD method to rate base fuel as a high-deposit fuel and to show the efficacy of additives Y and Z. Under these conditions, it would be expected that additives W and X would also be effective and that fuel B would give lower deposits than fuel A.

It is noted that the test configuration (vertical ISD) is physically very similar to the GDT configuration (Figure 2) but provided the much lower air flow rate desired based on the "additive volatility" theme.

C. Conclusions

Bench test conditions were sought to simulate the driving cycle thought to emphasize MPFI fouling, i.e., 15-minute urban driving followed by a 45-minute soak period. In the bench test, a deposit tube is kept at a constant temperature to simulate the "hot soak period." To simulate injector fuel weepage (leakage) during the "hot soak period," test fuel is sprayed at a controlled rate onto the hot deposit tube. After a sufficient

test period (spraying fuel onto the hot tube for time periods ranging from 1 to 6 hours) to provide weighable deposit on the tube, the deposit tube is washed with fuel or n-heptane at room temperature to simulate the washing of the injector-metering orifice during the driving section of the vehicle test cycle.

Based on selected test criteria for the high deposit fuel A and intermediate deposit fuel B, a set of optimized test conditions were established, using the apparatus described in SAE Paper No. 790204 with the carburetor sleeve section removed. This configuration is referred to as the Gasoline Detergency Test (GDT). The test conditions arrived at during the first phase of this program included a test time of 3 hours with main airflow at 30.5 L/min, fuel flow of 4 mL/min, no acid flow, and aluminum deposit tube at 300°F. Under these conditions, fuels A (high-deposit Ashland Batch No. 4), B (intermediate deposit Phillips J), C (low-deposit Indolene Clear), and additives W, X, Y, and Z in fuel A (at two concentrations H for high and L for low) were tested for repeatability and additive efficacy. Data showed:

- Base fuels are ranked for deposit tendencies repeatably based on calculated standard deviation values
- Fuel A with additives gave repeatable results based on standard deviation
- Additives W and X at H and L concentrations were effective in reducing deposit levels (below that of base fuel A) as measured by n-heptane-washed deposit weights
- Additives Y and Z gave repeatable deposit levels greater than the high deposit base fuel A at both H and L concentrations for Y and H concentration for Z
- Additives Y and Z at the H concentration in intermediate fuel B resulted in increased deposit levels using Y but not Z
- Additive Y had neutral effect in fuel C, giving very low values as did fuel C. These data suggested that the volatility of additive Y (and by inference, perhaps, additive Z) was involved in the lack of efficacy of additive Y in fuel A as opposed to thermal or oxidative additive instability.

Since all four additives are assumed to be effective in vehicle tests, temperature and air flow rates were varied to find conditions at which additives Y and Z (in fuel A) provided a deposit soluble in n-heptane or the test gasoline itself. Based on

promising data at very low air flow rates and/or lower temperatures, it was theorized that additive volatility (while not disallowing thermal and/or oxidative stability) was a critical property of additives Y and Z. Design of the GDT apparatus does not lend itself to good repeatable operations at low air flow rates due to cone nozzle design. A system utilizing airless spray technology was suggested but not evaluated.

Tests with the (Federal Test Method 500.1) ISD apparatus show that the volatility of additives Y and Z (at the H concentration in fuel A) can be overcome at 300°F, using 300 mL of test fuel followed by cooling and then washing with test fuel. Use of 275°F test temperature showed improved efficacy of additives Y and Z, especially when oriented in a vertical rather than horizontal spray position. The latter modified procedure used fuel washing after cooling to room temperature and showed fuel A to give high-deposit levels comparable to that of the GDT procedure. The ISD apparatus operated in this configuration is very similar to the GDT configuration, but affords the use of much lower air flow rates, a flat fuel spray, and improved baffle arrangement.

It is noted here that in bench tests of this type, the deposit levels may not be the most useful information for a user to judge a fuel's propensity for forming MPFI deposits. The deposit behavior during fuel-washing in the test procedure may be the key to commercial fuel screening for acceptability when considered in conjunction with the magnitude of deposit formed. In other words, observation that the deposit dissolves into the fuel during washing (independent of whether total deposit removal occurs) may form the basis for judging a gasoline acceptable for commercial use. When no dissolution is observed (indicating the absence of additives), then the magnitude of deposit weight would determine utility.

D. Recommendations

For the GDT apparatus, it is recommended that:

- Compare GDT data results to MPFI-fouling results being developed for CRC at EG&G for fuel A and additives W, X, Y, and Z (at the L concentration).
- If additives Y and Z are shown to be effective additives, then consider development of optimized repeatable procedure at main air flows much lower than 30.5 L/min rate using 250°F or 300°F tube temperature.

- Optimized condition should consider use of test fuel (followed by n-heptane) for tube deposit washing (rather than only n-heptane) after tube has cooled to room temperature and without the 60-minute after-test deposit tube heating.

Depending on the degree of correlation found for the GDT data and the MPFI-fouling data, it is recommended:

- That the modified ISD (Federal Test Method 500) operated in a vertical spray position be evaluated for test repeatability and that results at 275° be compared to the CRC MPFI-fouling test results.

III. APPROACH II: BOMB METHOD

A. General

Rather than attempting to modify an existing method to measure the MPFI deposit-forming tendencies of gasolines, the Bomb Method was developed to simulate more closely those conditions which result in deposits being formed on injector tips in service. The most "successful" driving cycle that results in injector fouling requires a 15-minute operating period, followed by a 45-minute soak period. A number of observations and studies carried out earlier by various researchers have suggested MPFI fouling is exacerbated under the following conditions:

- **Fuel slowly but continuously coating the injector pintle during the soak period.** It is generally agreed that slow-weeping injectors foul much more readily than non-weeping or high-weeping injectors.
- **Elevated temperatures or thermal gradients in the vicinity of the injectors.** Road tests and driving cycles for creating deposits have shown that engine temperatures must be sufficiently high after engine shutdown to provide adequate heat during the soak period.
- **Slow laminar flow of fuel on the metal surface of the pintle, followed by exposure to sufficiently high temperatures in alternate cycles.**

Although certain metals may offer a catalytic surface, stainless steel is known to be extremely inert. Thus, the stainless steel pintle is not believed to be a contributing factor to the kinetics of any reaction that may be occurring. Since pressure in the port is close to atmospheric, pressure was also eliminated as a possible requirement for fouling to occur.

B. Experimental

To provide uniform heat in a closed system, a Parr Bomb Calorimeter was modified to accommodate a variety of probes as illustrated in Figure 11a. The Parr Bomb was chosen because it is a readily available shelf item that can provide high temperatures in a safe environment. Initially, five holes were drilled into the device to accommodate two thermocouples, a "sleeve" holder, a spray tip, and an intake air tube.

As reported in an earlier progress report, efforts to measure deposits from atomized fuel were unsuccessful. The weights of washed deposits were inconsistent. This lack of consistent results was largely attributed to erratic spray patterns on the surface of the steel sleeve. Thus, it was decided to abandon the spray technique and to revise the delivery system so that the process occurring on injector tips could be more closely simulated.

As a result, the apparatus was subsequently modified to achieve the following objectives:

- Deliver a given amount of fuel slowly across a metal test surface for 15 minutes.
- Allow all the fuel to remain on the metal test specimen during a 45-minute "soak" cycle.
- Utilize a delivery system designed to deliver a measurable amount of fuel while carrying out the two steps above in alternate cycles.

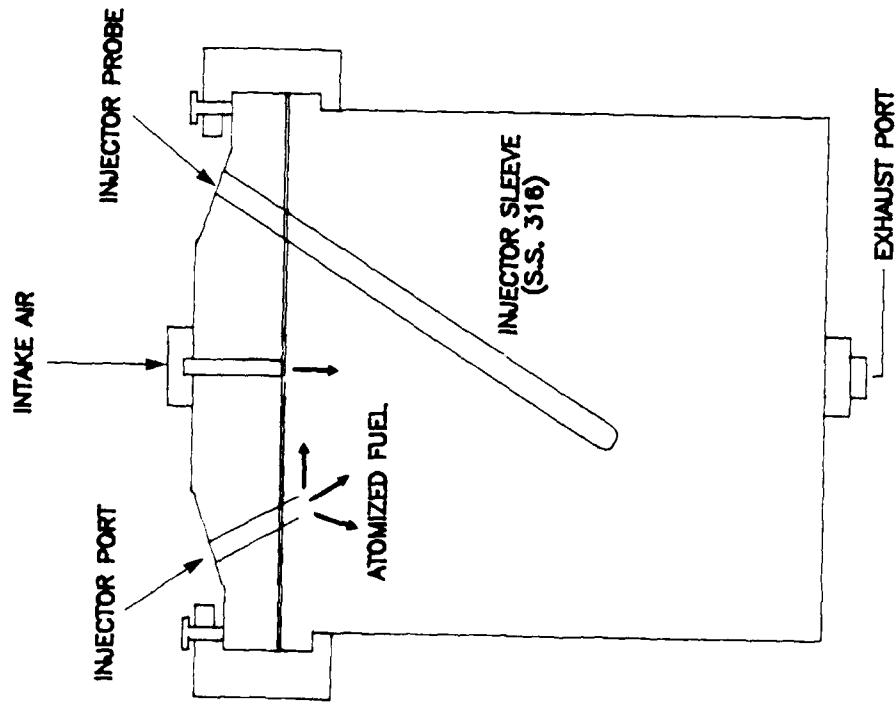


FIGURE 11a. SCHEMATIC OF FIRST GENERATION BOMB APPARATUS

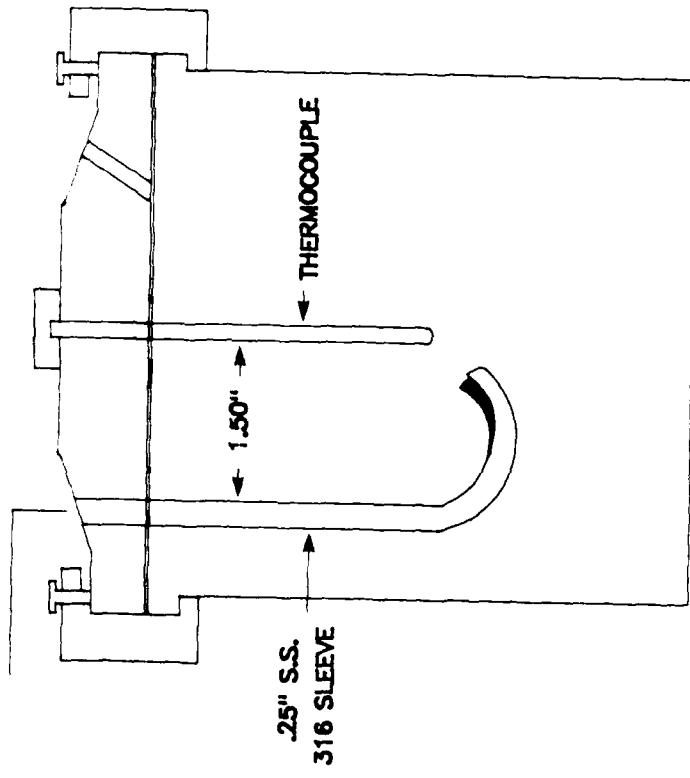


FIGURE 11b. SCHEMATIC OF MODIFIED BOMB APPARATUS

The schematic in Figure 11b illustrates the modifications introduced in order to achieve these objectives. As seen in the schematic, a "J" sleeve was prepared by cutting a 6-in. stainless steel tube so as to form a trough for the fuel delivered by a variable syringe pump. As described in an earlier progress report, the experiments were performed as follows:

The test fuel was added to the "J" tube by using a 6-in. stainless steel needle attached to a 5-mL syringe positioned in a syringe pump as shown in Figure 12. The variable syringe pump was set to deliver fuel at 0.07 mL per minute so that approximately 1 mL of fuel was slowly added over a 15-minute period. All the fuel delivered at this rate remained in the trough during the 15-minute soak cycle. Temperatures in the bomb were maintained at $290^{\circ}\text{F} \pm 5^{\circ}$ during the cycle. This cycle consisted of adding 1 mL of fuel and allowing to soak for 20 minutes. The cycle was repeated until 5 mL of fuel were added to the tube. Ambient pressure was maintained in the system by allowing vapor to escape from the delivery tube and through an additional small opening in the top of the bomb; temperatures were measured using two thermocouples that were placed in the center of the bomb approximately 2.5 cm from the J tube.

The "J" tube did not perform as well as anticipated. Gasoline delivered to the tube had a tendency to settle in the trough and did not always evaporate completely. Hence, it was decided to increase the surface area of the test specimen to allow complete evaporation of the fuel.

A cylindrical configuration was designed in order to optimize the surface area, thereby accommodating a greater volume of fuel. Several 0.040-in. holes were drilled through the neck of an aluminum cylinder to allow the fuel to cover the 1-in. O.D. X 4-in. long outer surface of the cylinder as shown in Figure 13. Schematics for the final modifications used throughout the remainder of the project are shown as Figures 12 and 14.

Initial experiments using the cylinder produced reasonably good results. However, it was observed that, when tested as discussed previously, fuel occasionally accumulated at the bottom of the cylinder. To ensure complete coating of the cylinder surface without fuel dripping from the tip, compressed clean air was passed through the center of the bomb. This procedure assisted in volatilizing the fuel during the soak cycle. It was noted that passing air through the bomb at approximately 2 L/min would permit increasing the amount of fuel being tested. The rate of fuel and total volume could be increased from 0.07 mL/min and 5 mL to 0.1 mL/min and 9 mL without fuel reaching the tip of the cylinder.

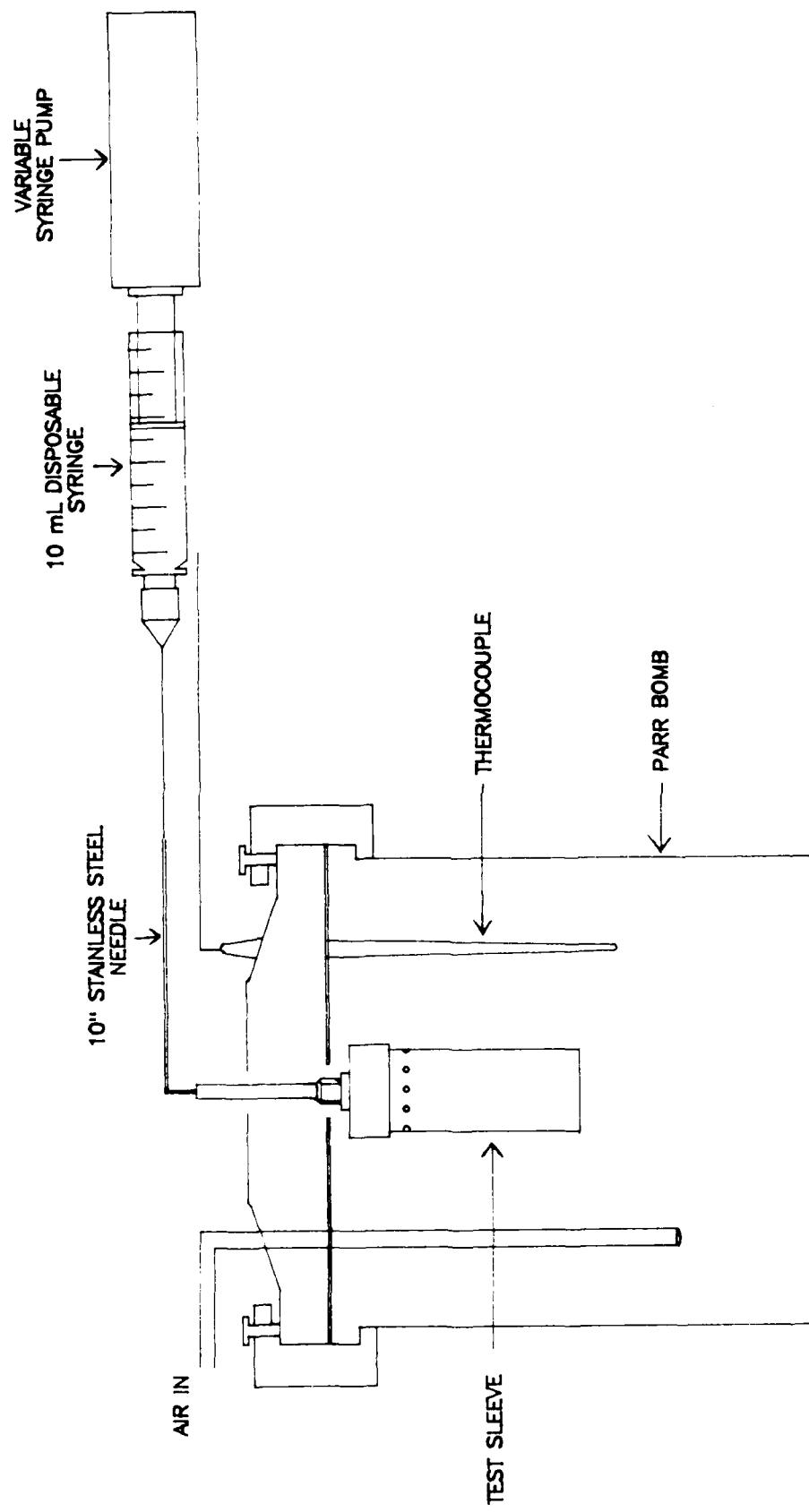


FIGURE 12. SCHEMATIC OF MODIFIED PARR BOMB AND VARIABLE FLOW SYRINGE PUMP

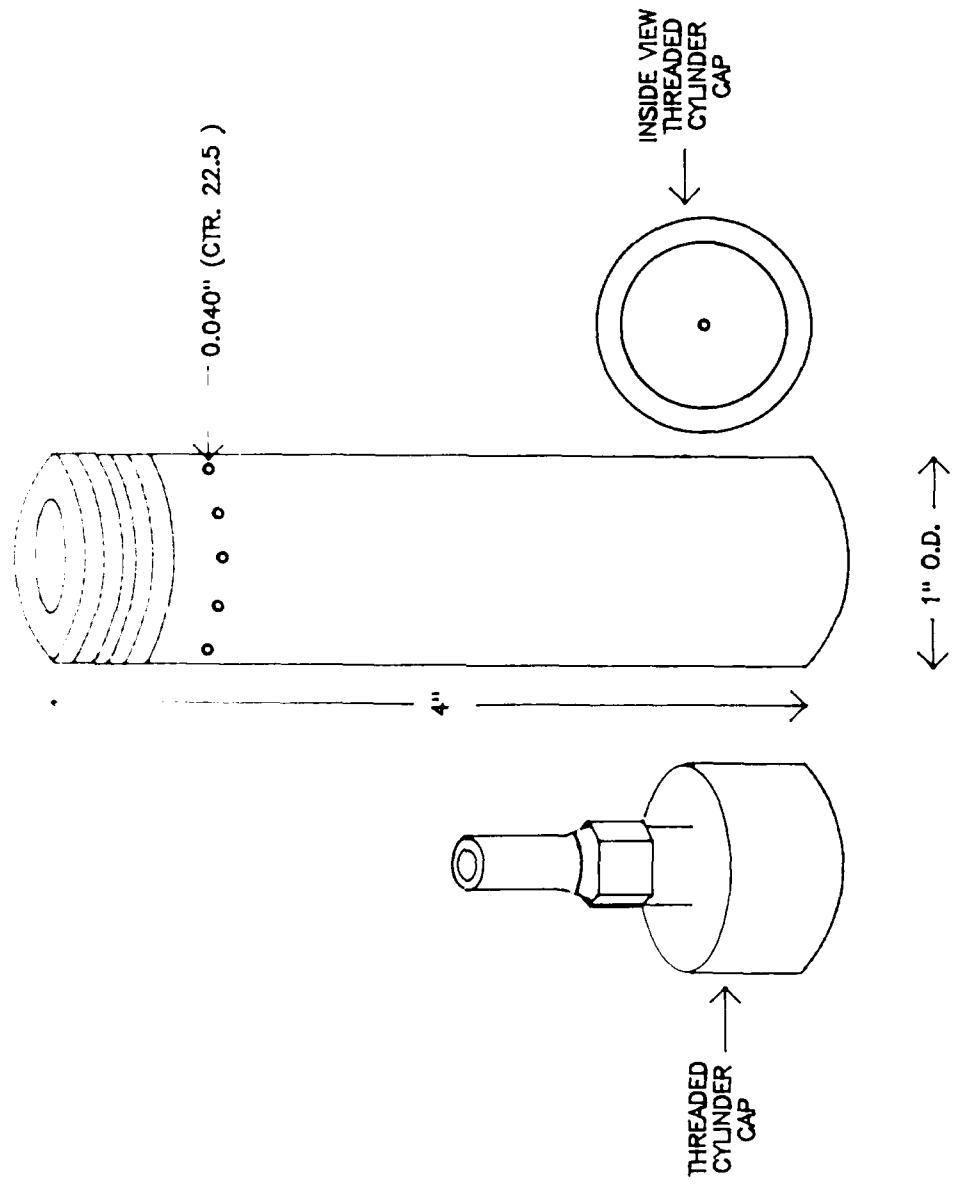


FIGURE 13. SCHEMATIC VIEW OF TEST SLEEVE AND CAP

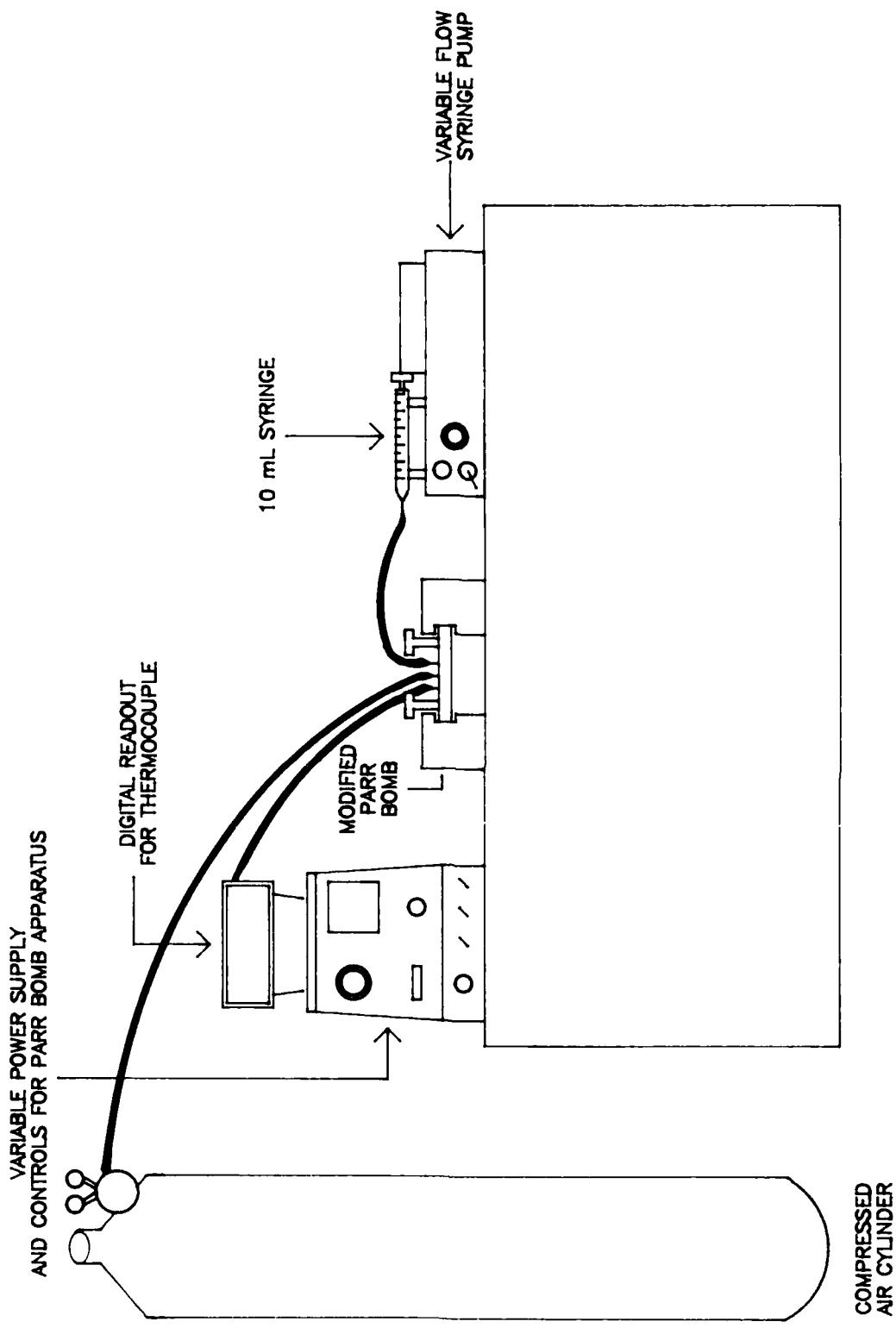


FIGURE 14. SCHEMATIC OF COMPONENTS ASSEMBLED FOR BOMB APPARATUS

Time and monetary constraints did not allow the study of additional parameters. Thus, it was decided to perform the experiments under the following conditions:

- 1) The aluminum cylinders were scrubbed vigorously with a scouring pad, and followed with washings of heptane/toluene (70/30) and acetone. The cylinders were then placed in an oven at 150°F for 0.5 hour and, afterward, cooled in a desiccator.
- 2) The test cylinder was placed in the bomb and the power was turned on to heat the system. Then a disposable 10-mL syringe fitted with a 10-in. stainless steel needle was charged with 9 mL of the test fuel. The filled syringe was positioned in the syringe pump holder with the needle tip leading into the top of the test sleeve.
- 3) Upon reaching 300°F ± 5°, the syringe pump was turned on, and fuel was delivered at a rate of 0.1 mL/min for 15 minutes.
- 4) After 15 minutes, the syringe pump was turned off. Then, 2 L/min of compressed, bottled air was passed through the apparatus to the center of the bomb.
- 5) After 30 minutes of "soaking," the pump switch was turned on to begin delivering fuel to the cylinder.
- 6) After delivering a total of 9 mL of fuel to the test surface and allowing for the last "soak" cycle, the bomb was turned off and cooled to about 150°F. The bomb cover was removed, and the cylinder was placed in a desiccator.
- 7) Upon reaching room temperature, the cylinder was weighed, and then washed three times in three 300-mL beakers containing 250 mL of heptane/toluene (70/30). The cylinder was then transferred to a 250°F oven for 0.5 hour (or overnight).
- 8) Upon drying, the cylinder was removed from the oven. It was then placed in a desiccator, allowed to cool to room temperature, and then weighed to the nearest 0.1 mg. Weights are reported as "unwashed" and "washed."

Although the experimental procedure as described above can be carried out in approximately 7 hours, the time frame can probably be reduced considerably. For example, some time-consuming lab techniques such as placing the cylinders in a desiccator during the drying procedure may not be necessary.

C. Results and Discussion

The results from evaluating Ashland Batch 4 (fuel A) and Indolene (fuel C) by the Bomb Method are presented in Tables 12 and 13. The date of each test is listed

TABLE 12. CRC FUEL INJECTOR DEPOSIT TEST RESULTS ON ASHLAND FUEL

Date	Fuel	Bomb	Air	Fuel	Fuel	Deposits, mg/9 mL	Specimen
		Temperature, °F, $\pm 5^\circ$	Flow Rate, 1./min	Flow Rate, mL/min	Volume, mL	Unwashed	
6-10-87	Ashland PFI	300	1.3	0.10	9	21.9	Cylinder
6-11-87	Ashland PFI	300	2	0.10	9	24.0	Cylinder
6-12-87	Ashland PFI	300	2.5	0.10	9	11.1	Cylinder
6-13-87	Ashland PFI	300	2	0.10	9	10.6	Cylinder
6-16-87	Ashland PFI	300	2	0.10	9	9.2	Cylinder
6-17-87	Ashland PFI	300	1.5	0.19	9	7.3	Cylinder
6-24-87	Ashland PFI	300	2	0.10	9	12.9	Cylinder
6-25-87	Ashland PFI	300	2	0.10	9	11.7	Cylinder
6-26-87	Ashland PFI	300	2	0.10	9	16.5	Cylinder
7-14-87	Ashland PFI	300	2	0.10	9	14.5	Cylinder
9-01-87	Ashland PFI	300	2	0.10	9	14.6	Cylinder
9-04-87	Ashland PFI	300	2	0.10	9	8.3	Cylinder
9-09-87	Ashland PFI	300	2	0.10	9	9.0	Cylinder
9-01-87	Ashland-Drum II	300	2	0.10	9	3.4	Cylinder
9-02-87	Ashland-Drum II	300	2	0.10	9	7.6	Cylinder
9-09-87	Ashland-Drum II	300	2	0.10	9	7.5	Cylinder
9-01-87	Ashland-Drum I	300	2	0.10	9	10.5	Cylinder
9-11-87	P65-8-87 PFI	300	2	0.10	9	6.3	Cylinder
9-11-87	P65-8-87	300	2	0.10	9	9.3	Cylinder
9-16-87	Ashland-Drum III	300	2	0.10	9	9.8	Cylinder
9-16-87	P65-8-87	300	2	0.10	9	9.0	Cylinder
9-17-87	Ashland-EG&G	300	2	0.10	9	9.1	Cylinder
9-17-87	Ashland-EG&G	300	2	0.10	9	9.8	Cylinder

TABLE 13. CRC FUEL INJECTOR DEPOSIT TEST RESULTS ON INDOLENE

Date	Fuel	Bomb Temperature °F, ± 5°	Air Flow Rate, L/min	Fuel Flow Rate, mL/min	Fuel Volume, mL	Unwashed Deposits, mg/9 mL	Washed Deposits, mg/9 mL	Specimen
6-24-87	Indolene	300	2	0.10	9	9.7	0.3	Cylinder
6-25-87	Indolene	300	2	0.10	9	8.6	0.3	Cylinder
6-26-87	Indolene	300	2	0.10	9	15.8	3.0	Cylinder
6-29-87	Indolene	300	2	0.10	9	13.1	0.4	Cylinder
7-01-87	Indolene	300	2	0.10	9	6.8	0.6	Cylinder
9-04-87	New Indolene	300	2	0.10	9	10.3	0	Cylinder
9-09-87	New Indolene	300	2	0.10	9	11.2	0.1	Cylinder
9-11-87	New Indolene	300	2	0.10	9	7.5	0.4	Cylinder
9-15-87	New Indolene	300	2	0.10	9	12.5	0.6	Cylinder
9-16-87	New Indolene	300	2	0.10	9	12.7	0.9	Cylinder

in the first column while the operating conditions, total amount of fuel tested, unwashed and washed deposits, and test specimen are all listed in corresponding columns.

The washed deposits are classified as "permanent" deposits. It is assumed that these deposits would eventually build up on injector tips, while unwashed deposits are considered fuel soluble.

The fuel labeled PFI was taken from a 55-gallon drum received from Ashland in January and labeled Drum II. In March 1987, approximately 2 gallons were transferred to a hexane-washed can, and were stored in the lab at ambient temperatures until September 1987. Drum III, which was also received in January, remained sealed at temperatures of 36°-38°F until 1 gallon was taken from the drum on September 1, 1987. The Drum I sample was received in December and, while several samples were drawn from it, the drum remained sealed at 38°F. The sample designated P65-8-87 was obtained from a 6,000-gallon Ashland fuel delivery received in August 1987; Ashland-EG&G was presumably shipped to EG&G sometime during August. As seen by the corresponding dates in the first column, the quantity of deposits obtained from the Ashland fuel dropped considerably beginning about the first week in September (9-1-87) when compared to deposits obtained earlier in the program.

After noting the drop in washed deposits from the PFI sample (9-01-87) and a low value from Drum I (9-01-87), a test on freshly drawn fuel from Drum II was run on 9-02-87. From this test, only 0.2 mg of washed deposits were obtained. A repeat of the PFI sample on 9-04-87 resulted in only 0.2 mg of washed deposits. Although the drop in washed deposits appears to vary from sample to sample, the results are reasonably consistent for samples tested in duplicate except for the PFI sample, which was re-run on 9-11-87 and one of the tests on Drum III. The results of the duplicate runs on samples from Drum II, P65-8-87 and Ashland-EG&G, nevertheless, were reasonably consistent.

Results from running Indolene, which was used as a control, are presented in Table 13. The terms "Indolene" and "New Indolene" were given to Indolene on hand for the early part of the program and a new batch obtained later in the project. Except for one test on 6-26-87, the washed deposits data were consistently below 1 mg.

Tests carried out on additives W, X, Y, and Z are presented in Tables 14 through 17. At both clean-up and keep-clean concentrations, measurable deposits were quite small compared to the base fuel. These small values suggested that "permanent" washed deposits were not allowed to form on the test cylinders. Moreover, measurable washed deposits averaged less than indolene deposits, particularly with tests using additives W and Y.

The data presented on Tables 14 through 17 represent tests carried out on additive-treated Ashland fuel. As reported earlier, A signifies Ashland fuel, while W, X, Y, and Z represent additives from four different distributors (2 amine-type and 2 polymeric-type additives). Finally, the H and L in the code signify recommended clean-up concentrations (H for high) or keep-clean concentrations (L for low). Very low values for washed deposits were measured from the Ashland fuel treated with either high or low concentrations of any of the additives.

Serial dilutions of Ashland fuel treated at keep-clean concentrations were prepared by diluting the original additive-treated fuels (AWL, AXL, AYL, and AZL) one-to-one with the PFI base fuel. For example, AWL was diluted 1:1 by simply blending 50 percent of AWL with 50 percent of the original PFI Ashland fuel. That 50/50 blend was then diluted to 1:3 by blending the 50 percent sample 1 to 1 with the PFI base fuel, resulting in a sample containing 25% additive-treated fuel and 75% PFI Ashland base fuel. The final dilution contained approximately 3 percent of the additive-treated fuel (AWL) and 97 percent of the PFI base fuel.

As seen in Table 14, deposits for additive W appeared to begin increasing at about a 1/8 concentration level. Except for one sample, a definite increase in washed deposits was observed at 1/16 and 1/32 concentration levels.

The same distinct trend was not observed with additives X, Y, and Z as shown in Tables 15, 16, and 17. However, there was some indication of deposit build-up at lower concentrations for additive X.

As pointed out earlier, some concern was expressed when the drop in washed deposits was observed on 9-1-87, shortly after the dilution studies were carried out. The

TABLE 14. CRC FUEL INJECTOR DEPOSIT TEST RESULTS FOR EVALUATION OF ADDITIVE W

Date	Fuel	Bomb Temperature, °F. ± 5°		Fuel Flow Rate, mL/min		Fuel Volume, mL	Deposits, mg/9 mL		Specimen
		300	2	0.10	9		8.8	0.3	
7-07-87	AWH	300	2	0.10	9	9	10.2	0.2	Cylinder
7-09-87	AWH	300	2	0.10	9	9	6.3	0.1	Cylinder
7-16-87	AWH	300	2	0.10	9	9	6.3	0.1	Cylinder
7-09-87	AWL	300	2	0.10	9	9	9.6	0.2	Cylinder
7-16-87	AWL	300	2	0.10	9	9	7.0	0.1	Cylinder
7-28-87	AWL (50%)	300	2	0.10	9	9	7.0	0	Cylinder
8-11-87	AWL (50%)	300	2	0.10	9	9	9.7	0.1	Cylinder
7-29-87	AWL (25%)	300	2	0.10	9	9	8.8	0	Cylinder
8-06-87	AWL (25%)	300	2	0.10	9	9	8.8	0.7	Cylinder
8-11-87	AWL (25%)	300	2	0.10	9	9	5.6	0.1	Cylinder
7-30-87	AWL (12.5%)	300	2	0.10	9	9	8.9	0	Cylinder
8-06-87	AWL (12.5%)	300	2	0.10	9	9	9.3	1.3	Cylinder
8-11-87	AWL (12.5%)	300	2	0.10	9	9	11.0	0.7	Cylinder
7-31-87	AWL (6.25%)	300	2	0.10	9	9	15.2	0	Cylinder
8-06-87	AWL (6.25%)	300	2	0.10	9	9	9.6	1.4	Cylinder
8-13-87	AWL (6.25%)	300	2	0.10	9	9	11.6	1.4	Cylinder
8-07-87	AWL (3.125%)	300	2	0.10	9	9	9.6	1.4	Cylinder
8-13-87	AWL (3.125%)	300	2	0.10	9	9	8.5	1.4	Cylinder

TABLE 15. CRC FUEL INJECTOR DEPOSIT TEST RESULTS FOR EVALUATION OF ADDITIVE X

<u>Date</u>	<u>Fuel</u>	<u>Bomb Temperature, °F, ±5°</u>	<u>Air Flow Rate, L/min</u>	<u>Fuel Flow Rate, mL/min</u>	<u>Fuel Volume, mL</u>	<u>Deposits, mg/9 mL Unwashed</u>	<u>Deposits, mg/9 mL Washed</u>	<u>Specimen</u>
					<u>mL</u>	<u>Washed</u>	<u>Specimen</u>	
7-10-87	AXH	300	2	0.10	9	7.8	0.7	Cylinder
7-15-87	AXL	300	2	0.10	9	6.9	0.4	Cylinder
7-28-87	AXL (50%)	300	2	0.10	9	8.4	0.6	Cylinder
8-13-87	AXL (50%)	300	2	0.10	9	8.6	0.2	Cylinder
7-29-87	AXL (25%)	300	2	0.10	9	7.8	0	Cylinder
8-17-87	AXL (25%)	300	2	0.10	9	9.1	0.3	Cylinder
7-30-87	AXL (12.5%)	300	2	0.10	9	11.9	0	Cylinder
8-17-87	AXL (12.5%)	300	2	0.10	9	9.5	0.5	Cylinder
7-31-87	AXL (6.25%)	300	2	0.10	9	18.4	0.2	Cylinder
8-17-87	AXL (6.25%)	300	2	0.10	9	10.0	0.8	Cylinder
8-19-87	AXL (3.125%)	300	2	0.10	9	10.2	0.8	Cylinder

TABLE 16. CRC FUEL INJECTOR DEPOSIT TEST RESULTS FOR EVALUATION OF ADDITIVE Y

Date	Fuel	Bomb Temperature, °F., +5°	Air Flow Rate, L/min	Fuel Flow Rate, mL/min	Fuel Volume, mL	Unwashed Deposits, mg/g mL	Washed Deposits, mg/g mL	Specimen
7-08-87	AYH	300	2	0.10	9	5.6	0.1	Cylinder
7-09-87	AYH	300	2	0.10	9	16.1	0.2	J-Sleeve
7-07-87	AYL	300	2	0.10	9	8.4	0.6	Cylinder
7-09-87	AYL	300	2	0.10	9	8.5	0	Cylinder
7-16-87	AYL	300	2	0.10	9	9.9	0.1	Cylinder
7-28-87	AYL (50%)	300	2	0.10	9	7.1	1.1	Cylinder
8-19-87	AYL (50%)	300	2	0.10	9	7.3	0.2	Cylinder
7-29-87	AYL (25%)	300	2	0.10	9	10.9	0.8	Cylinder
8-07-87	AYL (25%)	300	2	0.10	9	7.4	0.2	Cylinder
8-19-87	AYL (25%)	300	2	0.10	9	9.3	0.5	Cylinder
7-30-87	AYL (12.5%)	300	2	0.10	9	10.9	1.3	Cylinder
8-04-87	AYL (12.5%)	300	2	0.10	9	7.4	0.3	Cylinder
7-31-87	AYL (6.25%)	300	2	0.10	9	10.5	0.5	Cylinder
8-04-87	AYL (6.25%)	300	2	0.10	9	9.5	0.3	Cylinder
8-07-87	AYL (3.125%)	300	2	0.10	9	7.0	0.4	Cylinder

TABLE 17. CRC FUEL INJECTOR DEPOSIT TEST RESULTS FOR EVALUATION OF ADDITIVE Z

Date	Fuel	Bomb Temperature, °F, ±5°	Air Flow Rate, L/min	Fuel Flow Rate, mL/min	Fuel Volume, mL	Deposits, mg/g mL Unwashed	Deposits, mg/g mL Washed	Specimen
7-10-87	AZH	300	2	0.10	9	5.3	0.2	Cylinder
7-15-87	AZH	300	2	0.10	9	3.9	0.3	Cylinder
7-10-87	AZL	300	2	0.10	9	6.9	0.4	J-Sleeve
7-14-87	AZL	300	2	0.10	9	7.1	1.1	Cylinder
7-28-87	AZL (50%)	300	2	0.10	9	12.5	0.7	J-Sleeve
7-29-87	AZL (25%)	300	2	0.10	9	12.2	0.5	J-Sleeve
7-30-87	AZL (12.5%)	300	2	0.10	9	9.5	0.4	J-Sleeve
7-31-87	AZL (6.25%)	300	2	0.10	9	11.4	0.4	J-Sleeve

question arises as to whether the additives were still effective at greatly diluted concentrations, or the fuel aged to the extent that deposits were considerably reduced.

The only evidence that the PFI fuel was still producing deposits observed earlier in the program is the additive dilution data on AWL, where deposits begin to increase on 8-6-87 to 8-13-87 at a dilution factor of 12.5 percent.

Due to the design of the test cylinder and the delivery system, it was observed that fuel did not always flow uniformly through all the holes to the outside surface of the cylinder. This uneven flow was evidenced by uneven lacquer deposits on the cylinder surface and around the cap threads. The inability to deliver all of the fuel uniformly no doubt accounted for much of the inconsistent data obtained for the washed deposit weights.

Due to the possible similarities of the unwashed and washed deposits measured in the bomb method and existent gums, there was some concern that perhaps "gums" were simply being evaluated on the metal cylinder analogous to the ASTM D 381 method. The data for washed gums by D 381 shown in Table 18 were obtained at SwRI as well as tests reported earlier by Ashland. As seen in Table 18, washed gum values for fuel A showed an increase of approximately 60 percent in weight as a function of time despite the differences in storage and time frame between analysis.

TABLE 18. WASHED GUM VALUES FOR BATCH 4 FUEL
(Test Date) mg/100 mL

<u>SwRI</u>		
Fuel A ₁ *	(8-13-87)	4.3
	(3-24-87)	6.8
Fuel A ₂ **	(1-16-87)	4.0
Fuel A ₃ ***	(4-07-87)	9.7
<u>Ashland</u>		
	(8-14-86)	6.0
	(8-14-87)	9.8

* Fuel A₁ - Drum II -- received in January, stored at room temperature

** Fuel A₂ - Drum III -- received in January, stored at 38°F

*** Fuel A₃ - Stored at ambient outdoor temperatures from October 1986,

If the two highest (14.8 and 22.0 mg/9 mL) and lowest (0.2 and 0.2 mg/9 mL) values are discarded and the values for washed deposits run from 6-12-87 through 9-01-87 are averaged, the washed deposits would total about 50 mg/100 mL. Hence, compared to a washed gum value from Ashland fuel of about 5 to 7 mg/100 by D 381, the washed deposits by the Bomb Method are about nine times greater. It should also be noted that as washed gum values by D 381 increased as a function of time, which would be anticipated, washed deposit values by the bomb method decreased. This observation strongly suggests that the deposits observed by the bomb method are distinctly different from existent gums present in fuel A. It further suggests that a chemical reaction on the cylinder surface must be taking place with certain species present in fuel A. Moreover, it may also be concluded that the particular species undergoing chemical transformation in fuel A diminish as a function of time and perhaps storage temperature, while existent gums increase.

The lack of reproducible data obtained by the Bomb Method can no doubt be attributed to the method of applying fuel to the surface of the cylinder. By creating a dead volume space above a newly designed cylinder to ensure uniform, quantitative application of fuel to the surface, it is anticipated reproducible results can be obtained by the Bomb Method.

D. Summary and Conclusions

A novel approach for measuring MPFI injector deposit-forming tendencies of fuel has been evaluated. By slowly coating a cylindrical metal surface with a thin film of gasoline and applying sufficient amounts of heat in alternate cycles of coating and "soaking," two critical requirements of deposit formation were utilized.

The deposit generated from a known injector-fouling fuel (fuel A) were measured gravimetrically and compared to a relatively "clean" fuel (fuel C). Though some difficulty with reproducibility was evident, significantly greater concentrations of deposits were measured from fuel A when compared to fuel C. Fuel A was treated with two different types of additives--polymeric and amine—from four different manufacturers at high, clean-up levels and low, keep-clean concentrations and evaluated by the Bomb Method. Very few deposits were measured by any of the additive-treated fuels at high or low treatment levels.

Greatly reduced levels of each additive in fuel A were tested for "additive response" by measuring deposit formation as a function of additive concentration. Several additives appeared to begin losing their efficacy at lower concentrations in fuel A. The lack of reproducible results has been attributed to uneven coating of the test cylinder with fuel as a result of the design of the delivery system. The approach and methodology are believed to have a great deal of merit. With an improved design of the delivery system and optimization of experimental parameters, the Bomb Method promises to be a viable bench test method for measuring the deposit-forming tendencies of fuel.

E. Recommendations

The results presented herein, in addition to observations made while developing the Bomb Method, suggest that additional work will be required to optimize the test parameters for measuring the injector deposit tendencies of fuels. It is anticipated that critical evaluation of all the variables in the method will result in improvements upon the reproducibility and accuracy of the method. It is recommended, therefore, that the following modifications and/or studies be carried out in efforts to perfect the Bomb Method:

1. Redesign both the cylinder and delivery system such that a given aliquot of fuel completely covers the exposed outer surface uniformly.
2. Vary temperatures in the bomb in 10°C increments from 80°C (175°F) to 150°C (300°F) in order to find the optimum temperature for deposit formation.
3. Study the effects of exposure and flow rate using gases such as oxygen or sulfur dioxide in addition to compressed air upon deposit formation.
4. Vary the finish of both stainless steel and aluminum test cylinders to study the effects, surface porosity may have upon deposit formation.
5. Attempt to procure and evaluate several "fresh," commercial fuels that range from mild to severe in MPFI deposit-forming tendencies.

6. After all the parameters have been optimized, several additives will be evaluated using the same commercial fuels to determine additive response or deposit formation versus additive concentration.
7. Distill the deposit-forming fuels in 10-percent recovery increments and evaluate ten distillation cuts to determine which fraction(s) are involved in deposit formation.
8. Carry out preparative column chromatography to isolate aromatic, olefinic, and saturate-rich fractions to determine which class of compounds is responsible for injector fouling.

Although additional observations and experimental findings may present other opportunities to perfect the bomb method, it is anticipated that the foregoing studies will provide sufficient information and data to result in a simple, direct, rapid, inexpensive, and effective bench test for screening fuels with a tendency to form injector deposits.

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